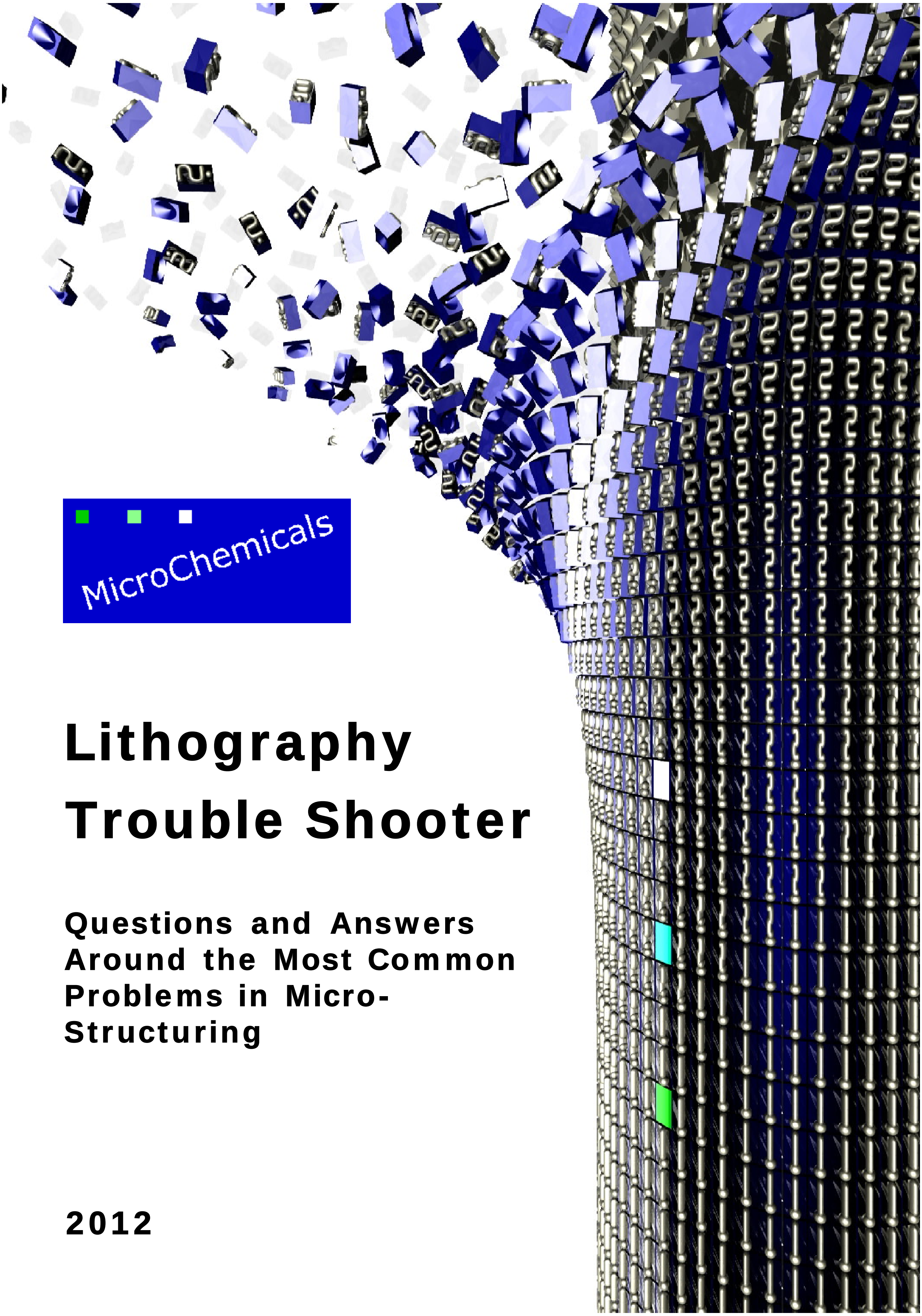


MicroChemicals

# Lithography Trouble Shooter

## Questions and Answers Around the Most Common Problems in Micro-Structuring

2012

Dear Reader,

The launch of a litho-process from concept to optimized result as well as the running production process often has to overcome several obstacles much in the same way as a hurdler encountering each hurdle.

This Trouble Shooter addresses potential problem areas and provides answers to the most frequently asked questions in the field of photolithography and microstructuring. We have kept the explanations as compact as possible in order to maintain maximum clarity. More detailed information is given in our booklet *Photolithography 2012*.

## How to Use the Lithography Trouble Shooter

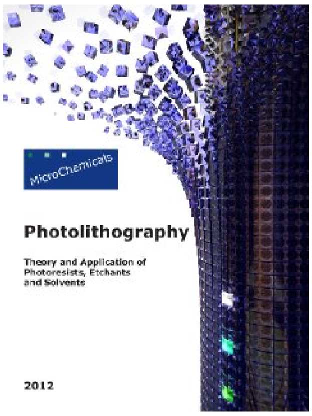
This booklet contains 16 chapters with each one focusing on one specific topic followed by possible solutions to problems one may encounter. The bold-faced links (e. g. „ 5.4) refer to a specific section in our present booklet *Photolithography 2012* giving more detailed information with plots and graphics for a better understanding.

Details concerning certain products and their fields of application can be found in our catalogue of goods *Our Products 2012*.

If you would like one or more complimentary copies of our brochures, please contact us as at *brochure@microchemicals.net* - thank you for your interest!

Wishing you much success,

Your MicroChemicals Team



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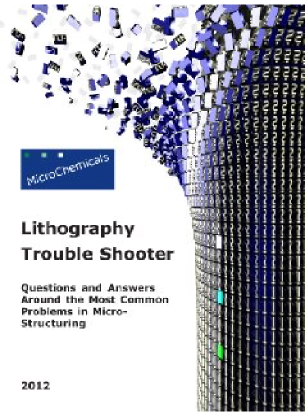
Photolithography

Theory and Application of Photoresists, Etchants and Solvents

2012



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Lithography Trouble Shooter

Questions and Answers Around the Most Common Problems in Micro-Structuring

2012

MicroChemicals

Our Products

Photoresists, Developers, Removers, Ancillaries, Solvents, Etchants, Yellow Light and Wafer

2012

# Content

## A) *Strange Appearance of the Photo Resist*

### 1. Discolouring?

i) Positive and image reversal resists darken over months, accelerated at elevated storage temperatures. The photo active compound forms azo-dyes, having a strong absorbance in the visible part of the spectrum without any impact on the UV sensitivity. The discolouration proceeds very slowly and therefore is often only noted when switching to a new resist lot. „ 3.6

ii) The contact of the resist with water or unsuited solvents such as isopropyl alcohol, or a freezing of the resist may change its colour. In this case, a deterioration of the resist can not be excluded. „ 3.6

iii) On different substrate materials, the resist film will show a different colour.

iv) Especially in case of thin resist films, a change of the resist film thickness of only few 10 nm will cause an interference-based colour change of the resist film. Such a thickness change can be induced by solvent loss, resist ageing, changed temperature or air humidity, or modified coating equipment or –parameters.

v) The attack of $HNO_3$-containing etchants or high baking temperatures can cause a brownish discolouring of the resist structures.

### 2. Uncoated Substrate Areas after Resist Coating?

i) An inferior resist wetting/adhesion (on noble metals or insufficiently cleaned substrates, on $SiO_2$-substrates treated with an HF-dip with incomplete $SiO_2$ removal, or at a high ambient air humidity) promotes the appearance of uncoated substrate areas. „ „ 4.2 - 4.6

ii) A too small resist volume can also be the reason for uncoated areas on the substrate. Depending on the resist viscosity, the desired resist film thickness, and the substrate size, 1 - 5 ml resist per substrate is recommended. Dispensing the resist as central as possible will improve the result. „ 5.4

iii) In case of smooth substrates, a high acceleration (> 1000 rpm/s) towards the final spin speed suppresses the appearance of uncoated parts on the substrate. „ 5.4

iv) Textured substrates may require a two-step spin profile: After spreading a comparable high resist volume at low spin speeds (e. g. 1000 rpm) for a few seconds, ramping upwards to the final spin speed adjusts the desired resist film thickness. „ 5.4

v) Bubbles and particles are often the starting point for defects in the coated resist film, as detailed in the following section. „ 5.5

### 3. Bubbles and Comet-Like Structures after Spin Coating?

i) Air bubbles in the resist incorporated by transport, refilling or dispensing of the resist, often act as starting point of defects in the resist film. A delay before dispensing of – dependant on the resist viscosity – a few hours will help for outgassing the bubbles. „ 5.5

ii) $N_2$-bubbles originate from the gradual decomposition of the photo active compound in the liquid resist. If – after storage for a certain time – the resist bottle is opened, the $N_2$ dissolved in the resist may form bubbles. A delay before dispensing of – dependant on the resist viscosity – a few hours will help for outgassing the bubbles. Another $N_2$ source is nitrogen used in the dispense system to transport the resist from the bottle to the substrate. „ 5.5

iii) Particles impact on the resist film homogeneity in a similar way as air- or $N_2$-bubbles do. Beside insufficiently cleaned substrates or an inferior clean room class, particles often stem from an insufficient substrate cleaning or an expired resist. A further resist dilution or elevated storage temperatures will accelerate resist ageing and thus particle formation. „ 5.5

### 4. Rough or Bumpy Resist Surface after Coating?

i) Expired resists or resists stored under the wrong (temperature) conditions, as well as resists diluted too strongly or diluted with unsuitable solvents, may form particles. In early stage averaging, the coated resist reveals a silky resist surface. Strongly deteriorated resist may also show bigger lumps and coating failures. Please contact us for recommended storage conditions and a suitable thinner! „ 3.1 - 3.6

ii) Besides particle formation, gelification is another irreversible ageing effect promoted by elevated storage temperatures, and high resist dilution ratios. The coated resist film shows a glossy, but bumpy surface.

## B) Resist Coating Techniques: Common Problems

### 1. Spin Coating: Edge Bead?

i) Spinning a highly-viscous resist (e. g. the AZ® 4562 or 9260 for 5 - 30 µm resist film thickness, or the ultra-thick resist AZ® 40 XT for films > 30 µm) at high spin speed helps to suppress the edge bead.

ii) Circular substrates: Manual edge bead removal at approx. 500 rpm with AZ® EBR Solvent (not acetone or other low-boiling solvents!) dispensed onto the edge of the rotating substrate, immediately followed by a spin-off of the solvent at an elevated spin speed. „ 5.3

iii) In the case of resist film thicknesses exceeding the standard thickness of a certain resist: An elevated spin-speed (e. g. 2000 - 4000 rpm) for few seconds with steep ramps up and down, instead of low spin speeds for a longer period of time. „ 5.3

iv) A ‘spin-off’ of the edge bead by abruptly increasing the spin speed at a certain stage of spin coating: The resist film should be solvent-poor enough to prevent further thinning, while the edge bead needs a viscosity still low enough for proper spin-off. For this purpose, the optimum spin profile has to be found individually. „ 5.3

v) A multiple coating with an elevated spin speed for each coating cycle requires a sufficiently high-viscous resist as well as a short softbake between two coating steps in order to prevent the dissolution of the already coated resist film by the next one. „ 5.3

vi) For thick or solvent-rich resist films, a delay between coating and softbake prevents the edge bead from growing during softbake due to the thermally reduced viscosity. The delay time depends on the resist film thickness and can be shortened by applying moderate temperatures (e. g. stepwise: room temperature ... 50°C ... 95°C). „ 5.3

vii) An assembly on the spin-coater substrate holder with a cut-out which tightly holds the substrate as close inlay.

viii) Edged substrates: If applicable, removing (breaking) the outer pieces of the substrate bearing the edge bead; alternatively manual cleaning of the substrate from the edge bead with cleanroom wipes and solvent. „ 5.3

ix) In the case of positive or image reversal resists, an exposure and development of the edge bead is also possible but may cause T-topping of the resist profile due to the impact of the developer on the unexposed resist. „ 5.3

### 2. Spin Coating: Comet-Shaped Structures?

i) These radial resist inhomogeneities often stem from air or $N_2$-bubbles, more rarely from particles, both driven outwards during spin coating. Possible reasons and work-arounds are listed in A)3.i) - iii). „ 5.4, 5.5

### 3. Spin Coating: Substrate Partially Uncoated after Spin-Coating?

i) Possible reasons and work-arounds are listed in A)2. „ 5.4, 5.5

### 4. Spray Coating: Inferior Edge Coverage?

i) If the concentration of especially high-boiling solvents in the resist is too high, the growing resist film will flow and leave the edges of the texture uncoated. Please consult us for suited dilution recipes for spray coating resists! „ 6.3, 6.4

ii) Modifications in the equipment (e. g. an increased distance between nozzle and substrate) or the spray coating parameters (e. g. and increased rate or pressure) may increase the average solvent concentration of the resist droplets landing on the substrate. As a result, the growing resist film will flow and leave the edges of the texture uncoated. „ 6.3, 6.4

### 5. Spray Coating: Rough Resist Film?

i) A concentration of especially high-boiling solvents in the resist being too low prevents the droplets landing on the substrate from flowing and thus smoothing the resist film. Please consult us for suited dilution recipes for spray coating resists! „ 6.3, 6.4

ii) Modifications in the equipment (e. g. a reduced distance between nozzle and substrate) or the spray coating parameters (e. g. a reduced rate or pressure) will decrease the average solvent concentration of the resist droplets landing on the substrate preventing a sufficient smoothing of the resist film. Please consult us for suited dilution recipes for spray coating resists! „ 6.3, 6.4

### 6. Dip Coating: Inhomogeneous Resist Film Thickness?

i) A resist film thickness increasing from the upper (concerning the position during coating) towards the lower part of the substrate points towards the wrong solvent composition of the resist: If the resists need too much time to sufficiently increase its viscosity immediately after coating, the resist will drain off during dip coating. Optimized dip coating resists (such as the *MC Dip Coating Resist*) have a solvent composition allowing very homogeneous resist film thicknesses. „ 7.4

ii) Vibrations by the step motor or from the environment cause rather strong variations of the resist film thickness. If the substrate mounting comes into contact with the liquid resist, striations caused by resist draining off may deteriorate the coating homogeneity. A non-constant air flow around the cuvette is another possible reason for inhomogeneities in the resist film thickness. „ 7.3

### 7. Roller Coating: Inhomogeneous Resist Film

i) Resists optimized for roller coating often have thixotropic properties. The viscosity changes when mechanical stress is applied making the resist film thickness more homogeneous as compared to the usage of standard resists for roller coating application.

### 8. Screen Printing: Suited Resists

i) The mechanical properties of photo resists make them less suited for screen printing. If nevertheless this coating technique should be applied, we recommend tests with resists with a high viscosity such as AZ® 4562, AZ® 9260 or AZ® 40 XT.

## C) Exposure Techniques: Occasional Problems

### 1. Laser Scribing

i) Bubbles or cracks after exposure in positive or image reversal resist films very probably stem from the high $N_2$-generation rate during the photoreaction. Using resists with a low photo active compound concentration such as AZ® 4562 or AZ® 9260, or AZ® 40 XT without any $N_2$ release during exposure or lowering the total resist film thickness as well as increasing the softbake time/temperature might help. If the bubbles or cracks still appear, the laser intensity should be decreased, or the whole laser scribing process split into several steps to allow the $N_2$ to outgas in time.
„ 12.3, 12.10

ii) If the exposed resist cannot be developed despite a sufficient exposure dose, or neighboured resist parts finally not be removed, the resist film has probably become too hot during exposure. Hereby the resin of the resist thermally crosslinks thus becoming insoluble in solvents or alkaline removers. If the laser intensity cannot be decreased, please try the steps listed in Section P).

### 2. Laser Interference Lithography

i) This technique generally requires very thin resist films, often realized via dilution of existing resists. While the AZ® 1500 or 6600 resists are known to form particles in the case of high dilution ratios, AZ® 701 MiR and the AZ® ECI 3000 resists are capable for a strong dilution for resist films of 200 nm and less. As a thinner, we recommend the solvent PGMEA = *AZ® EBR Solvent*.
„ 3.5

ii) Laser Interference Lithography often requires resists with a high resolution potential in the sub-µm range. For this purpose, we recommend the thermally stable AZ® 701 MiR for applications such as dry etching or lift-off, or the AZ® ECI 3000 series for wet chemical processes requiring a superior resist adhesion.

### 3. Exposure Wavelengths Beyond/Below the Standard Resist Absorption.

i) Wavelengths exceeding the absorption maxima of resists by several 10 nm (e. g. > 450 nm for broadband resists) are generally suited for exposure, since the resist absorption does not abruptly end at a certain wavelength. However, the low absorption with penetration depths much higher than the resist film thickness require high exposure doses. Additionally, the weak absorption promotes the lateral propagation of incident light in case of rough, textured, or transparent substrates thus lowering the attainable spatial resolution.
„ 2.4

ii) Too small wavelengths (< 340 nm) only theoretically improve the resolution. Due to the low penetration depth of the incident light, and the parasitic absorption of the already exposed photo active compound, the exposure does required strongly increases. In combination with the short-wavelength-promoted Rayleigh-scattering, the attained resolution often decreases as compared to i-line exposure.
„ 2.4, 12.9

### 4. Sticking Between Resist Film and Mask

i) A softbake too cool/too short keeps the remaining solvent concentration in the resist film too high, which promotes sticking to the photo mask. We recommend a softbake at 100°C (hotplate) for one minute/µm resist film thickness.

ii) Particles on the mask promote the local sticking of the resist film. Cleaning the mask will help in this case.

iii) A hard-contact during exposure promotes the sticking between resist film and mask. If applicable, switching to the soft-contact mode will help.

iv) An inferior resist adhesion promotes the resist peeling from the substrate by the photo mask. Section F) gives information on resist adhesion improvement.

## D) Development: Minor Development Rate

### 1. Sufficient Rehydration?

i) DNQ-based positive resists (not AZ® 40 XT) and image reversal resists require a certain water content to allow a reasonable high development rate. After the softbake or image reversal bake, the now missing water has to diffuse back from the air into the resist film requiring a delay time between baking and exposure to rehydrate the entire photo resist film towards the substrate. The required rehydration time strongly depends on the resist film thickness and increases from approx. 1 min for 1 µm films to several hours for films exceeding 30 µm. „ 10

ii) In case of a sufficiently long rehydration time, the ambient air humidity impacts on the later development rate which significantly drops if the humidity is below 40 %. We recommend to keep the humidity constant at approx. 40 - 50 %. „ 10

### 2. Sufficient Exposure Dose in the Case of Positive Resists?

i) Generally, the required exposure dose depends on the resist, the resist film thickness, and the emission spectrum of the light source with respect to the resist to be exposed. We strongly recommend an exposure dose series and determine the development rate as a function of the exposure dose when starting with new resists or processes or after changes of the exposure equipment. „ 12.2

ii) In case of thin resist films (thickness < light penetration depth), an exposure dose being too low results in a constant but low development rate. Since a large fraction of the light reaches the substrate, the optical properties (reflection and transmission coefficient) strongly impacts on the required exposure dose. „ 12.7

iii) In case of thick resist films (thickness > light penetration depth), an exposure dose being too low results in a normal development rate until a certain depth where the development almost stops. „ 12.7

### 3. Compatible Developer?

i) Some photoresists require special developers for constant fast and residual-free development. For example, the AZ® 4500 series (AZ® 4533 and 4562) require the KOH-based AZ® 400K or the TMAH-based AZ® 826 MIF. For the negative resists AZ® nLOF 2000, we recommend the TMAH-based developers AZ® 326 MIF, 726 MIF or 826 MIF. The AZ® 111 XFS requires the developer AZ® 303. „ 34.1

### 4. Expired Developer?

i) $CO_2$-absorption from air limits the lifetime of open developer baths depending on the surface/volume ratio of the developer bath. A $N_2$ curtain reduces $CO_2$-absorption and thus decelerates the developer neutralisation. Even well-closed containments are permeable for $CO_2$, therefore the developer activity may decrease after the expiry date. „ 3.7

ii) A developed resist concentration too high also exhausts the developer: As a rule of thumb, approx. one per mille resist dissolved in typical developers significantly reduces the development rate. „ 15.5

### 5. Developer Dilution too High?

i) The KOH-based AZ® 400K and NaOH-based AZ® 351B are typically applied in a 1 : 4 dilution with water. AZ® Developer can be applied as a concentrate (high development rate), or in a 1 : 1 dilution (improved selectivity). The

TMAH-based ready-to-use developers AZ® 326 MIF, 726 MIF and 826 MIF are generally applied undiluted. The AZ® 303 is typically applied at a dilution ratio of 1 : 4 (for the AZ® 111 XFS) or 1 : 5 - 1 : 10 (for other resists). In case of very thin resist films or very small resist structures, increasing the dilution ratio by a factor of up to 1.5 above the ratios mentioned, sometimes make the process more stable but strongly lowers the development rate. „ 15.3

### 6. HMDS applied correctly?

i) In case of spin-coating of HMDS, a comparable thick HMDS film forms on the surface. Additionally, HMDS vapours from the spincoater will penetrate the resist film during resist coating. During the subsequent softbake, this excess of HMDS releases ammonia which diffuses into the resist and chemically modifies the resin near the substrate, and also may cause crosslinking of the resin. As a result, the development rate decreases, and through-development may become impossible (scumming). We strongly recommend to apply HMDS from the vapour phase onto heated substrates, and keep resist and resist film away from HMDS vapour. „ 4.8

ii) Many substances often used in cleanrooms such as ammonia or chlorobenzene interact with the chemistry of the photoresist. Diffusing into the resist film via the gaseous phase, during a subsequent baking step (e. g. softbake) the resist will become photochemically inert or crosslink thus reducing the development rate.

### 7. Thermal Decomposition of Photo Active Compound?

i) The photosensitive compound of photoresists is partially decomposed during baking steps. If the softbake temperature and -time are higher than recommended, the development rate decreases. We recommend a softbake temperature of 100°C for 1 minute/µm resist film thickness. „ 9.4, 9.5

ii) If the resist is stored beyond its expiry date, or at temperatures exceeding the recommended storage temperature, the photo active compound will gradually decompose thus reducing the attained development rate. Adjusting the exposure dose, developer concentration, or development time can compensate this effect to a certain extent. „ 3.6

### 8. Resist Film Thickness Higher than Expected?

i) The airflow conditions in the spin-coater impact on the evaporation rate of the solvent from the resist film coated. Therefore, any changes in the equipment (covered chuck) or substrate size and shape are a possible reason for changes in the attained resist film thickness.

ii) The ambient solvent saturation in the spin coater depends on the number of substrates coated before, and the time span between the last cleaning of the spin coater and the last resist coating. Since the atmosphere solvent concentration impacts on the solvent evaporation rate from the resist film, it is recommended to coat some dummy wafers before starting with new coating series.

iii) The resist viscosity and thus the attained film thickness depends on the temperature, and the solvent concentration which gradually drops over the months if the resist bottles are frequently opened. „ 3.6

iv) Spray- and dip coating resists often contain also a low-boiling solvent such as acetone or MEK which evaporates much faster than the solvents used in standard resists. Thus, the solvent loss and therefore attained resist film thickness is sometimes more pronounced. „ 6.3, 7.4

### 9. Changed Substrate Reflectivity?

i) Especially in case of thin (e. g. < 2 µm) resist films, the effective exposure dose absorbed by the resist film depends on the substrate properties (reflection coefficient and scattering). For this reason, the exposure dose required for a certain resist film depends on the substrate material and surface. While metals such as Ag, Cr or Pt show a high reflectivity of short wavelength light, ITO absorbs most of the blue and UV part of the spectrum and quartz and glasses transmit the major part of these wavelengths.

### 10. Negative or Image Reversal Resists in Image Reversal Mode?

i) An image reversal bake or post exposure bake too hot/too long as well as a significantly too hot softbake cause thermal crosslinking of the resin also in the unexposed resist which thereby show a slower development rate.

ii) If also the resist part to be developed is unintentionally exposed to short wavelength light, crosslinking or the image reversal reaction take place which lowers the development rate or makes through-development impossible. Possible reasons for an accidental exposure are insufficient yellow light conditions, diffraction due to a gap between photomask and resist surface, light scattering in the resist film in combination with a high exposure dose, diffuse reflection from a rough substrate or a lateral guidance of the light by textured or UV-transparent substrates (detailed in E)6.i)). „ 15.3

### 11. Chemically Amplified Resists?

i) The chemically amplified AZ® 40 XT requires a post exposure bake to perform the photoreaction, otherwise the development rate is almost zero.

## *E) Development: Dark Erosion too High*

### 1. Proper Developer Dilution?

i) With increasing developer concentration, the dark erosion rate growths faster than the development rate. The KOH-based AZ® 400K and NaOH-based AZ® 351B are typically applied in a 1 : 4 dilution with water. AZ® Developer can be applied as a concentrate (high development rate), or in a 1 : 1 dilution (improved selectivity). The TMAH-based ready-to-use developers AZ® 326 MIF, 726 MIF and 826 MIF are generally applied undiluted. AZ® 303 is typically applied at a dilution ratio 1 : 4 (for AZ® 111 XFS) or 1 : 5 ... 1 : 10 (for other resists). For thick resist processing or the demand of very high throughput, the developer concentration can be increased by some 10 % which, however, reduces the selectivity. „ 15.3

### 2. Compatible Developers?

i) Certain developers such as AZ® Developer or AZ® 826 MIF reveal slightly higher dark erosion rates for photoresists. If these developers are not required by the applied resists, more selective developers are recommended. „ 34.1

### 3. Optimum Softbake Conditions?

i) A softbake too cool/too short keeps the remaining solvent concentration at high values, thus increasing the dark erosion rate in developers. We recommend a softbake temperature of 100°C for 1 minute/µm resist film thickness. When using an oven, one has to consider the time the substrates need to heat up to the desired temperatures. When using a hotplate, thick substrates or substrates with minor heat conduction, as well as strained/curved substrates may reduce the effective temperature in the resist film. „ 9.5, 9.6

ii) A softbake too extended (too hot/too long) will thermally decompose a significant amount of the photo active compound. Since the unexposed photo active compound acts as inhibitor in alkaline solutions, its loss increases the erosion in developers. „ 9.3, 9.4, 16.1

### 4. Decomposition of Photo Active Compound during Resist Storage?

i) If the resist is stored beyond its expiry date, or at temperatures exceeding the recommended storage temperature, the photo active compound will gradually decompose thus reducing the attained development rate. The unexposed photosensitive compound of photoresists is a solubility inhibitor in alkaline solutions, thus decreasing the dark erosion in developers. „ 3.2, 3.6

### 5. In Case of Image Reversal- or Negative Resists?

i) The reason of the image reversal bake after the 1st exposure of image reversal resists is to make the exposed resist insoluble in the developer. If the image reversal bake is too cool/too short, the solubility keeps at a rather high value leading to resist erosion of the exposed part during development. The recommended image reversal bake parameters depend on the image reversal resist. Please contact us for detailed information! „ 17.3, 17.4

ii) Negative resist such as the AZ® nLOF 2000 resists require a post exposure bake (PEB) to crosslink the exposed part of the resist. If the PEB temperature or –time are not sufficiently high, the degree of crosslinking keeps at low values, leading to a resist erosion during development. For the AZ® nLOF 2000 resists we recommend a PEB temperature of 110 - 120°C for 1 - 2 minutes if applied on a hotplate. „ 18.2

### 6. Accidental Exposure of Positive Resists?

i) If the exposure dose is too high, light scattering (Rayleigh scattering) in the resist film may expose parts of the resist close to the exposed areas, which hereby become soluble in developers.

ii) A gap between the resist surface and mask (intended or accidental via bubbles or particles in the resist film or the photomask, or an edge bead of the resist) will cause an interference pattern on the resist and thus widen the exposed structures. Additionally, reflections between resist surface and photomask laterally guide light. „ 12.5

iii) Textured or rough substrates reflect or scatter light to parts of the resist film which should keep unexposed. In transparent substrates, light can be guided laterally thus exposing resist from the rear side. In both cases, an adjustment of the exposure dose to values not higher than required can be reasonable. Additionally, a bottom-layer anti-reflective-coating such as the AZ® Barli can be beneficial. „ 11.2

iv) Most positive tone resists are also sensitive to short-wavelength visible light as emitted by fluorescent tubes or electrical bulbs, or day light transmitted through windows. Therefore, all light sources should be filtered by e. g. yellow foils absorbing wavelengths < 520 nm. „ 8.1

### 7. Second Development Step?

i) During development, the alkaline developer also penetrates the unexposed resist to a certain depth. If the rinse after development is not sufficiently long, the developer residuals concentrate in the resist when the water evaporates and forms a strong base with the water from a second development step. Such as base acts as stripper and is able to remove a layer of the resist film. If a second development step cannot be waived, much care has to be taken to sufficiently rinse with water after the first development.

## F) Inferior Resist Adhesion

### 1. Optimum Substrate Pretreatment?

i) In the case of clean substrates, baking at 120 - 140°C for several minutes is sufficient for the desorption of $H_2O$. At 150°C, the OH-bonds apparent on oxidized surfaces (Si with native oxide, $SiO_2$, glass, many metals) decompose thus further increasing the resist adhesion. „ 4.3

ii) Substrates contaminated with particles or organic impurities can be cleaned with a two-stage cleaning process: Acetone removes organic impurities, a subsequent rinse in isopropyl removes the contaminated acetone thus avoiding striations. „ 4.2

iii) The correct application of HMDS is very important in order not to further deteriorate the resist adhesion: HMDS should be applied from the gaseous state onto heated substrates, never from the liquid phase. „ 4.8

iv) After $SiO_2$-etching with HF (e. g. 'HF-dip'), the resist adhesion strongly depends on the thoroughness of $SiO_2$ removal: With $SiO_2$ completely removed, the now H-passivated Si-surface reveals a hydrophobic surface with a very good adhesion. Incomplete etching with remaining oxide results in a poor and non-reproducible adhesion, which can be restored with a bake at temperatures > 700°C. „ 4.5

### 2. Using HF-containing Etchants?

i) HF diffuses into the resist film and may lead to a large scale resist peeling either during the etching, or after the subsequent rinsing by resist swelling and large-scale etching of the resist covered substrate material. Work-arounds are i) increasing the resist film thickness (= barrier against HF), and the usage of buffered HF (BOE). „ 25.2

### 3. On Noble Metals?

i) The resist wetting and adhesion on noble metals (e. g. silver, gold) is often worse. In this case, resists such as AZ® 111 XFS, AZ® 1514 H or the ECI 3000 series may improve the adhesion. While organic adhesion promoters such as HMDS are not able to significantly improve resist adhesion, a metallic adhesion layer (titanium or chromium) results in a very good adhesion to resists. If required, such few nm thick metal films can easily be etched after development and/or after resist removal. „ 4.6

### 4. Optimum Softbake Parameters?

i) A softbake too cool/too short keeps the remaining solvent concentration too high with inferior resist adhesion as a consequence. In this case, especially thick resist films show $N_2$ bubble formation during exposure. We recommend a softbake temperature of 100°C for 1 minute/µm resist film thickness. When using an oven, one has to consider the time the substrates need to heat up to the desired temperatures. When using an hotplate, thick substrates or substrates with minor heat conduction, as well as strained/curved substrates may reduce the effective temperature in the resist film. A work-around are a longer softbake time or higher softbake temperature. „ 9.5 - 9.7

ii) A softbake too hot/too long embrittles the resist film and causes mechanical stress between resist and substrate. In this case - especially after abrupt cooling after the softbake - the resist film may loose adhesion to the substrate. In this case, a slow temperature ramp down might be beneficial.

### 5. Positive Tone Resists on Transparent Substrates?

i) Especially in case of too high exposure doses, light can be guided in transparent substrates and hereby expose the resist near the substrate. As a result, small resist structures may lift in the developer. „ 12.9

### 6. Image Reversal- or Negative Tone Resists?

i) Too low exposure doses or a post exposure bake being too cool/too short will keep the substrate-near resist soluble in the developer. This may cause lifting of especially small resist structures from the substrate during development. „ 17.3, 17.4, 18.1, 18.2

### 7. Double-Sided Metallized Substrates?

i) Substrates coated with different metals on both sides may cause a galvanic cell in aqueous solutions, accompanied by gas ($H_2$-) formation under the resist peeling it from the substrate. A protective coating with e. g. AZ® 520 D on the “rear side” of the substrate will help.

## G) Minor Resist Resolution

### 1. Suitable Photoresist?

i) Positive resists such as AZ® 1505, AZ® 1512 HS or AZ® 6612 allow structure sizes below 1 µm, AZ® 701 MiR and the AZ® ECI 3000 series even below 0.5 µm. If high-resolution negative tone resists are required, thin films of the AZ® nLOF 2000 series allow sub-µm features for e. g. lift-off application. „ 33

### 2. Gap between Photomask and Resist Surface?

i) Particles, bubbles in the resist film, or an resist edge bead may cause a gap between mask and resist which degrades the theoretical and achieved resolution by diffraction and light scattering. „ 12.5

### 3. Suited Softbake Parameters?

i) A softbake too hot/too long thermally decomposes a significant part of the photoactive compound DNQ of positive and image reversal resists. If not exposed, the DNQ acts as a solubility inhibitor in alkaline solutions, thus decreasing the dark erosion in developers. Since the development rate decreases at the same time, the contrast of the resist and hereby its resolution performance deteriorates. We recommend a softbake temperature of 100°C for 1 minute/µm resist film thickness. When using an oven, one has to consider the time the substrates need to heat up to the desired temperatures. When using an hotplate, thick substrates or substrates with minor heat conduction, as well as strained/curved substrates may reduce the effective temperature in the resist film. „ 9

ii) A softbake too cool/too short keeps the remaining solvent concentration too high with increased dark erosion as a consequence, which deteriorates the possible resolution of the resist film. The previous section informs about recommended softbake parameters. „ 9

### 4. Compatible and Sufficiently Diluted Developers?

i) Certain developers such as AZ® Developer or AZ® 826 MIF have higher dark erosion rates for photoresists than other developers, making them less suited for high-resolution demands. The AZ® 4500 series (AZ® 4533 and 4562) require the KOH-based AZ® 400K or the TMAH-based AZ® 826 MIF. For the negative resists AZ® nLOF 2000, we recommend the TMAH-based developers AZ® 326 MIF, 726 MIF or – better – 826 MIF. AZ® 111 XFS requires the developer AZ® 303. „ 34

ii) The dark erosion grows faster than the development rate when the developer concentration is too high. The section E)1.i) lists the recommended dilution ratios of common developers. For very small structures, the dilution ratio can be increased. „ 15.3

### 5. Optimum Exposure Dose?

i) Positive resists exposed with a too small exposure dose require for through-development either a prolonged development time, or a stronger developer concentration. In both cases the dark erosion increases thus deteriorating the possible contrast and, respectively, resolution. „ 12.2

ii) Negative or image reversal resists exposed with a too small exposure dose show an increased erosion of the exposed resist during development, which also reduces the attainable resolution of the resist. „ 17.3, 18.1

iii) If the exposure dose is too high, light scattering in the resist film also exposes 'dark' parts of the resist, which hereby become soluble (in case of positive resists), or insoluble (in case of image reversal or negative resists) in developers thus preventing the realization of very small structures. The section E)6 gives some more details on the possible reasons for accidental overexposure.

## H) Bubbles or Particles in Resist Film after Coating

### 1. Resist Bottles Moved?

i) Carrying or shaking resist bottles or moving them may inject small air bubbles into the resist. In this case, a delay before coating allows the bubbles to raise to the resist surface. The waiting time depends on the resist viscosity, 1 - 2 hours should be sufficient in most cases. „ 5.5

### 2. Resist Bottles Closed for a While?

i) Over months, the photo active compound of many resists releases $N_2$ which dissolves in the resist. When the resist bottle is opened, $N_2$ bubbles may form which - more or less slowly - raise to the surface. For this reason, shaking the closed bottle followed by a delay with the screw cap set loosely (pressure balance!) on the bottleneck is required to outgas the $N_2$. The waiting time depends on the resist viscosity, 1 - 2 hours should be sufficient in most cases. „ 5.5

### 3. Manual Resist Dispensing?

i) Dispensing the resist with pipettes often leads to air bubbles in the resist. As a work-around, give the resist-filled pipette some time allowing the air bubbles to raise away from the outlet. „ 5.5

### 4. Spray Coating?

i) If the fraction of a low-boiling solvent such as MEK or acetone is too high, the resist droplet may dry during flight and subsequently hit the substrate as a resist pellet. Chapter 6 gives details on the spray coating technique. „ 6.3

### 5. Expired Resist?

i) Expired resist or resist stored under wrong conditions may form particles. This effect especially concerns low-viscosity thin resists with a high concentration of the photo active compound. „ 3.6

### 6. Diluted Resist?

i) If a dilution is required, only suitable solvents (such as PGMEA or MEK) should be used, and a resist-specific maximum dilution should not be exceeded. Otherwise, the resist may rapidly form particles. Please contact us for suited solvents and dilution recipes! „ 3.5

### 7. Bumps in the Resist Film after Spin-Coating?

i) One or few mm-sized scattered resist structures on the resist-coated substrate often origin from a solidified resist in the dispenser nozzle. Especially in the case of a long delay after a previous dispensing step or a strong air flow (exhaustion), a resist skin forms in the nozzle and is scattered onto the substrate during the subsequent dispensing. Possible methods to inhibit the formation of such a resist skin between two dispensing steps are a shorter delay, a minimized or paused exhaustion or the pre-dispensing of a small resist volume beside the substrate.

ii) A cm-sized resist bump in the centre of the substrate (the location where the resist has been dispensed) often results from a too long delay between resist dispensing and the spin start. Hereby, the evaporation of the solvent from the resist solidifies especially high-viscous resists which no longer homogeneously thin out during spin-coating.

## I) Bubbles in Resist Film after Exposure

### 1. Optimum Softbake Parameters?

i) A softbake too cool/too short keeps the remaining solvent concentration too high allowing the $N_2$ formed during exposure to form bubbles in the resist film. We recommend a softbake temperature of 100°C for 1 minute/µm resist film thickness. When using an oven, one has to consider the time the substrates need to heat up to the desired temperatures. „ 9

### 2. Sufficient Resist Adhesion?

i) The $N_2$ generated during exposure of positive tone and image reversal resists may form bubbles at locations of minor resist adhesion to the substrate. Detailed information on substrate cleaning and adhesion promotion are listed in section F). „ 4

### 3. Exposure Intensity Too High?

i) The $N_2$ generated during exposure of positive tone and image reversal resists needs to dissipate from the resist film before its concentration becomes too high. If the exposure intensity is too high, the $N_2$ cannot timely outgas and therefore forms bubbles or resist cracks due to mechanical stress. Lowering the exposure intensity (= extending the exposure time), or splitting the exposure into several steps with delays in-between (especially reasonable in case of laser scribing) will help in this case. „ 13

### 4. Resist Film Thickness Too High?

i) Towards thicker resist films, it becomes more difficult for the $N_2$ formed during exposure to diffuse towards the resist film surface. Thus, bubble formation is much more pronounced during thick resist processing. For this reason, we recommend suited thick resists such as AZ® 4562 or 9260 with a lower photo active compound concentration causing less $N_2$ during exposure. „ 13

## J) Bubbles in Resist Film after Thermal Processes

### 1. After Baking Steps of Exposed Photoresist?

i) The $N_2$ generated during exposure of positive tone- and image reversal resists may form thermally activated bubbles in the softening resist during baking steps (post exposure bake or image reversal bake) or during any other process steps performed at elevated temperatures, such as electroplating. In this case, a delay between exposure and baking step will help.

### 2. After Coating (Sputtering, Evaporation) or Dry Etching?

i) Elevated temperatures may evaporate remaining solvent or water (from development) in the resist. In combination with the low ambient pressure, bubbles may occur. Increasing the softbake time or temperature will reduce the remaining solvent concentration, while a baking step after development reduces the water content. „ 19.2, 21.7

ii) A further reason for bubbling can be the undesired exposure of the resist film during evaporation/sputtering by thermal or recombination UV radiation with $N_2$ formation as a result. One work-around is the usage of image reversal resists in image reversal mode or a negative tone resist such as the AZ® nLOF 2000 series which do not release nitrogen during exposure. In case of positive tone resists, a flood exposure (without mask) of the developed resist structures with a certain delay to outgas the $N_2$ formed. „ 19.2, 21.7

## K) Wet Etching: Common Problems

### 1. Resist Adhesion Problems?

i) Possible reasons and work-arounds with respect to inferior resist adhesion are listed in section F). „ 25.1, 25.2

### 2. Underetching?

i) Isotropic etchants generally underetch the resist to an extent comparable to the etched depth. A more pronounced underetching points towards an inferior resist adhesion (s. section F) which - for the purpose of wet etching - often can be improved by a hardbake after development at approx. 140°C for 5 - 10 minutes. „ 25.1

### 3. Inhomogeneous Etch Start?

i) Even a few nm resist residuals on the substrate can act as a barrier against many etchants. Possible reasons for resist residuals are incompatible developers (D)3), incorrect HMDS treatment (section D)6.i)), or insufficient rinsing after development.

ii) Alkaline developers preferentially dissolve the (native) $Al_2O_3$ where the resist is primarily through-developed (at regions with lower resist film thickness, near the edges of cleared structures, or below cleared structures with larger features). Dependant on the extent of (desired or undesired) overdeveloping as well as delay between development and Al-etching, the process parameters may cause spatial inhomogeneous Al etching start. „ 23.1

### 4. Inhomogeneous Etch Depth during Al-Etching?

i) $H_2$ bubbles formed during Al-etching tend to stick to the substrate and hereby locally inhibit etching. These bubbles can be released either via ultrasonic treatment, or by repeatedly dipping the substrate into DI-water for 1 - 2 seconds between a certain number of etch cycles. „ 23.1

### 5. Resist Attack in $HNO_3$-Containing Etchants?

i) Strong oxidizing etchants attack the resist. A hardbake after development at 140 - 150°C improves the chemical stability of the resist. „ 16.1

### 6. Inferior Resistance against Alkaline (KOH, NaOH, TMAH ...) Solutions?

i) While a hardbake at 150°C or higher improves the alkaline stability of resists, cresol-based resists will never be stable enough to be suitable as an etch mask for KOH- or TMAH-based anisotropically silicon etching. For this purpose, we recommend the use of e. g. $SiO_2$ or $SiN_x$ hard masks.

## L) Dry Etching: Common Problems

### 1. Resist Rounding/Flowing?

i) If, during dry etching, the resist it heated above its softening point (110 - 130°C for most positive tone and image reversal AZ® resists), the resist profile starts rounding. Possible work-arounds are an optimized heat coupling of the substrate to its holder (e. g. some 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 anodized aluminium as rear infrared radiator) from the substrate holder, a reduced etch rate and/or multistage etching with cooling interval(s) in between, DUV-hardening, a thermally stable photoresist (e. g. AZ® 701 MiR or the AZ® 6600 series, or the AZ® nLOF negative resist for highest thermal stability as well as a sufficient softbake for a minimized remaining solvent concentration. „ 21.6

### 2. Bubbling of Resist?

i) Unexposed positive resists may be exposed with UV radiation present during dry etching. Thus, $N_2$ is released which can form bubbles in the thermally softened resist. „ 21.7

### 3. Resist Can Not Be Removed After Dry Etching?

i) High temperatures, DUV-radiation, and the ion bombardment may strongly crosslink the resist making its removal problematic. „ 21.6, 21.8, 28

## *M) Electro-Plating: Common Problems*

### 1. Resist Stability

i) AZ® and TI photoresists are stable in common acidic electroplating baths. „ 2.2, 20.2

ii) Aqueous alkaline solutions, however, attack the resist mask when the pH-value exceeds 10 (depending on the time and temperature of the bath). Moreover, one has to consider that the local pH-value near the metal deposition can be quite different from the value measured in the bath. Crosslinking resists such as AZ® 15 nXT, AZ® 125 nXT or the AZ® nLOF 2000 series are more stable in alkaline media as compared to positive and image reversal resists. „ 20.7

### 2. Inferior Resist Adhesion?

i) Resist swelling in the electro plating bath may reduce resist adhesion to the substrate. Section F) gives various hints for improving the resist adhesion. The negative resists AZ® 15 nXT and AZ® 125 nXT are optimized for electroplating and reveal a very good adhesion on most substrate materials. A thin titanium film (chromium or aluminium will also help) gives a very good adhesion promoter. If required, this film can be removed after development where the structures are cleared. In this case, care has to be taken not to underetch the adhesion layer under the resist film. „ 20.3, 4

### 3. Bath Contamination by Remaining Solvent?

i) Typical softbake parameters keep the remaining solvent concentration at approx. 2 - 4 %. If released to the electroplating bath, the solvent may impact the metal deposition chemistry with a reduced deposition rate or other problems as a consequence. In this case, a longer softbake and/or elevated softbake temperatures are recommended. „ 20.4

ii) Thick resist films will have a comparable high solvent concentration especially near the substrate even after a prolonged softbake. In this case, a baking step after development at temperatures of approx. 10°C below the resist softening point would be beneficial. „ 2.1, 9.6

### 4. Resist Profile

i) If steep resist walls are required, we recommend the AZ® ECI 3000 or AZ® 6600 resist series for resist film thicknesses of few µm, the AZ® 9260 for film thicknesses of 5 - 20 µm, or the negative resists AZ® 15 nXT or AZ® 125 nXT for thicknesses of 20 - 150 µm. „ 20.5

ii) A positive profile of the deposited metal structures requires a negative resist profile. This can easily be obtained with the AZ® nLOF 2000 negative tone resist allowing an adjustment of the undercut via the process parameters exposure dose and post exposure bake temperature. „ 18.4

iii) If the metal structures shall widen upwards, a positive resist profile is required. This can either be attained via a proximity gap of the photo mask during exposure, or a thermal reflow after development beyond the softening point of the photo resist used. „ 2.1

### 5. Bad Adhesion of the Metal Structures?

i) Some resists require special developers for residual-free development. If unsuitable developers are used, a few nm resist may stay on the substrate preventing a proper contact between substrate and deposited metal. Please consult Section D)3 for recommended developers. „ 34.1

ii) Incorrectly applied adhesion promoter may also deteriorate the contact between seed layer and deposited metal: HMDS applied form the liquid phase or on unheated substrates may crosslink the substrate-near resist which can not be developed. „ 4.8

iii) Crosslinking negative resists processed on certain metals (e. g. Cu is critical) crosslink under certain process conditions of the metallization and/or lithography, which stops the development some 10 or 100 nm before the substrate is reached. The negative resists AZ® 15 nXT and AZ® 125 nXT are Cu-compatible.

## N) Lift-Off Does Not Work (Well)

### 1. Optimum Coating Technique?

i) The more or less isotropic sputtering will also coat the resist sidewalls, while evaporation is much more directed and therefore generally gives better lift-off results, especially in case of thick (> several 100 nm) films.

### 2. Suitable Photoresist?

i) A pronounced undercut of the resist sidewalls makes the lift-off much easier as compared to perpendicular or even positive sidewalls. Therefore, negative tone resists such as the AZ® nLOF 2000 series, or image reversal resists such as AZ® 5214E or TI 35ES are a good choice. If nevertheless positive tone resists have to be used for lift-off application, the AZ® 6600 series or the AZ® 9260 thick resist allow almost perpendicular sidewalls. *Developer soaking* of positive resists may be an alternative - please ask us for details!

„ 17.8, 17.9

### 3. Resist Softening and Rounding during Coating?

i) During coating (evaporation, sputtering, CVD), the resist film may be heated by the evaporation source radiation, the condensing heat of the growing film, or the kinetic energy of the ions, above its softening point (110 - 130°C for most positive tone and image reversal AZ® resists). The resist profile starts rounding allowing the coating material to also cover the resist profile sidewalls. As a result, subsequent lift-off will work worse or become impossible. Possible work-arounds are listed in Section L)1.i). „ 19.1

### 4. Suitable Lift-Off Medium?

i) Using solvents with a high vapour pressure, such as acetone as a lift-off medium, sometimes promotes the re-adsorption of material already lifted onto the substrate. We recommend NMP as a lift-off medium, which can be heated up to 80°C if required. If necessary, ultrasonic treatment assists the lift-off process. „ 19.5

## O) Substrate Attack by Photochemicals

### 1. Discolouring / Attack by the Photoresist?

i) Under certain circumstances, additionally dyed resists such as PL 177 or AZ® 520D may stain especially porous substrates. In this case, the use of undyed resists should be considered.

ii) Resists usually contain rather strong organic solvents such as PGMEA which attack many polymers. A swift coating immediately after dispensing resist as well as a softbake immediately after the coating may help a little bit. Alternatively, a thin metal film protects the polymeric substrate from the solvent to a certain extent. The metal film can be etched after resist development and resist removal.

iii) Many resists form organic acids during exposure which might attack acidic sensitive substrates.

### 2. Attack by the Developer?

i) Aqueous alkaline (KOH-, NaOH or TMAH based) developers attack alkaline sensitive materials such as Al or ITO. *AZ® Developer* is optimized for low Al attack and, in an adjusted dilution, suited for almost all resists and processes. „ **34.1**

ii) Crosslinking negative resists such as the AZ® nLOF 2000 series, AZ® 15 nXT and 125 nXT can alternatively be developed in certain organic solvents. We recommend PGMEA = *AZ® EBR Solvent*.

### 3. Attack by the Remover?

i) AZ® 100 Remover is uncritical on alkaline sensitive materials as long as applied absolutely water-free. Even traces of water increase the pH-value to > 13 which attacks e. g. Al. „ **28**

ii) Undiluted NMP is pH-neutral, while the pH-value increase to approx. 10.5 when diluted with water in a 1 : 1 ratio. „ **28**

## *P) Photoresist Removal Does Not Work (Well)*

### 1. Organic Solvents as Removers?

i) Acetone, sometimes used as a stripper, promotes resist residuals on the substrate due to the fast evaporation. Heating acetone for faster stripping is absolutely not recommended due to the low flash point and high risk of fire. NMP (N-methyl-2-pyrrolidone) or the nontoxic DMSO (dimethyl sulphoxide) are much better lift-off media due to their low vapour pressure. In case of cross-linked resists, heating the NMP or DMSO up to 80°C may be required for resist removal. „ **28**

### 2. Alkaline Remover?

i) As an alternative to organic solvents, AZ® 100 Remover is optimized for stripping photoresists residual-free and striation-free. In the case of Al (-containing) substrates, even traces of water have to be avoided in order to prevent Al attack. NaOH or KOH (> 2%) can also be used as stripper (on alkaline stable substrates), a higher cross-linking degree may require higher concentrations and elevated temperatures. „ **28.3, 28.4**

### 3. Hardbake?

i) Negative tone resists, such as the AZ® nLOF 2000 series already crosslink during the post exposure bake (PEB) and crosslink more strongly at moderate hardbake temperatures. All cresol-resin-based AZ® resists thermally crosslink from approx. 150°C on. If applicable, lowering the hardbake temperature will improve the solubility of the resist in the remover. For heavily cross-linked positive resist films, we recommend the high-performance stripper TechniStrip® P1316 or – in the case of very thick resist films or Novolak-based negative resists – TechniStrip® NI555. „ **16.1**

### 4. After Coating (Evaporation, Sputtering, CVD)?

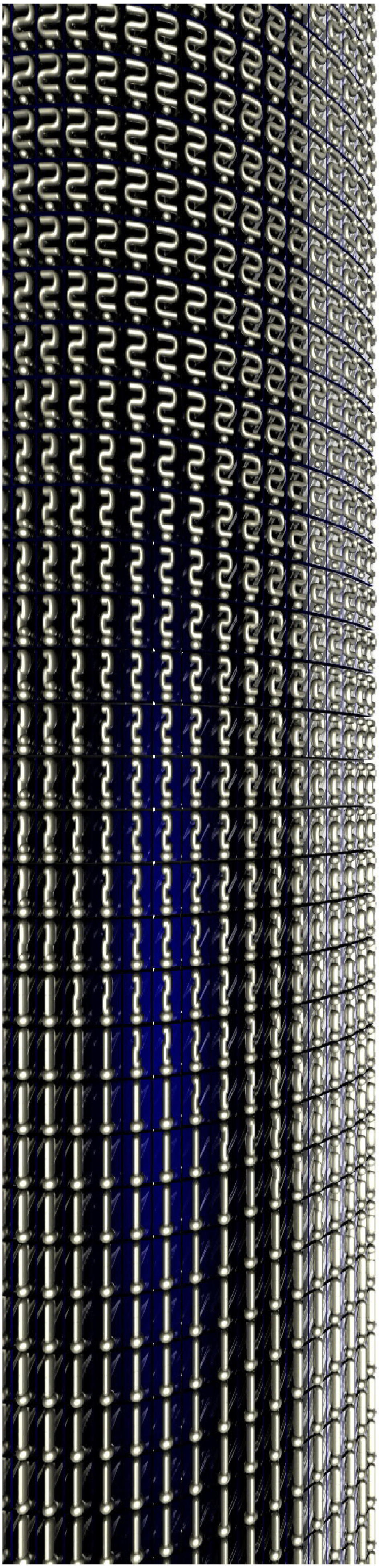
i) If the resist film is strongly heated by the evaporation source radiation, the condensing heat of the growing film, or the kinetic energy of the ions, cross-linking of the resist may decrease its removability. For heavily cross-linked positive resist films, we recommend the high-performance stripper TechniStrip® P1316 or – in the case of very thick resist films or Novolak-based negative resists – TechniStrip® NI555. Possible work-arounds for lowering the temperature during coating are listed in Section L)1.i). „ **19.1**

### 5. After Dry Etching?

i) Generally consider what is mentioned in the previous subsection. Additionally, re-deposition of material already etched onto the resist may prevent resist removal. In this case, the re-deposited material first has to be wet-chemically etched which can be assisted by ultrasonic treatment. „ 21.6

### 6. Crosslinked Negative Resists?

i) In the case of heavily cross-linking (e. g. by high process temperatures), Novolak-based negative resists such as AZ® nLOF 2000 or AZ® 15 nXT can be dissolved with TechniStrip® NI555, while for epoxy-based resists as well as dry films we recommend TechniStrip® P1316. TechniStrip® NI105 is a powerful remover especially designed for the ultrathick negative resist AZ® 125 nXT.



## Your Contact Persons

**Dr.-Ing. Christian Koch**

Tel.: +49 (0) 731 36080-409
Mobile: +49 (0) 178 7825198
Fax: +49 (0) 731 36080-908
E-mail: koch@microchemicals.eu

**Dr.-Ing. Titus J. Rinke**

Tel.: +49 (0) 731 36080-409
Mobile: +49 (0) 177 3332453
Fax: +49 (0) 731 36080-908
E-mail: rinke@microchemicals.eu

MicroChemicals

## Imprint

**Address**
MicroChemicals GmbH
Nicolaus-Otto-Strasse 39
Ulm, Germany 89079
Tel.: +49 (0) 731/36080 409
Fax.: +49 (0) 731/36080 908
E-mail: info@microchemicals.eu

**Managing Directors**
Dr.-Ing. Titus J. Rinke, Dr.-Ing. Christian Koch

**Place of Business** **VAT-ID**
Ulm HRB 4271 DE813168639