

Advancing greyscale lithography and pattern transfer of 2.5D structures using ma-P 1200G resist series

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ABSTRACT

Greyscale lithography is applied to manufacture complex 2.5D and freeform microstructures in photoresists. The thus obtained structures serve as master or template for different methods of pattern transfer into materials for final, permanent applications. Here, we describe the chemical background and processing fundamentals of typical positive photoresists used for this purpose as well as the characteristics of the ma-P 1200G resist series developed by us specifically for enabling greyscale lithography. Different resist patterning examples are presented as well as a method to transfer such 2.5D resist patterns by UV moulding into hybrid polymers to be used permanently.

INTRODUCTION

In greyscale lithography a photoresist layer is exposed with UV light of spatially modulated intensity, thereby controlling the amount of photons absorbed within the resist layer. The photoresist is chemically altered to different degrees depending on the exposure dose, inducing a gradual change of dissolution rates. During a wet development step this exposure dose and dissolution rate gradient is transferred into a film-thickness gradient of the photoresist layer remaining on the substrate. This way, complex 2.5D topographies with discrete or continuous height levels can be generated, which are highly relevant for the fabrication of diffractive, refractive and freeform microoptical elements, MEMS and MOEMS as well as microfluidics.

Particularly, greyscale lithography is applied when standard binary photolithography processes, including thermal reflow of binary structures to generate rounded patterns, does not suffice due to the complexity of the desired topography (Figure 1). This is the case for:

- Applications with high structure density, such as high fill-factor microlens arrays, where reflow patterns would merge
- Complex structures such as concave and convex lenses
- Combination of patterns at different length scales

- and hierarchical structures
- Microlenses or discrete diffractive patterns with different heights next to each other
- Any freeform microtopography used for e.g. beam-shaping and -steering in optical applications

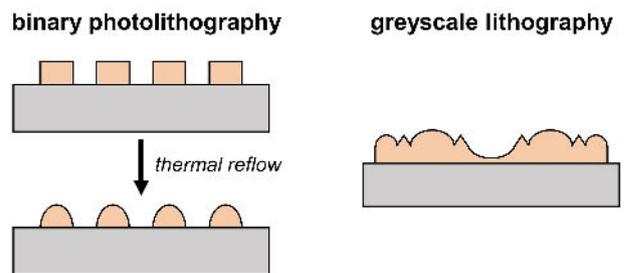


Fig. 1. Exemplary resist patterns manufactured with binary standard photolithography and thermal reflow, compared to greyscale lithography.

A general process scheme for greyscale lithography using a positive photoresist is shown in Figure 2. Individual steps are very similar to standard binary UV lithography comprising:

- Coating of the substrate with resist
- Softbake
- Relaxation and re-hydration of the photoresist film. The re-hydration time required increases exponentially with the film thickness, from seconds in thin films

- ($\leq 10 \mu\text{m}$) to 24 h or more for thick films ($\geq 50 \mu\text{m}$).
- Optionally, a short pre-exposure bake to stimulate a more homogenous water distribution in the resist film, especially in case of thick films which have been stored for a long time ($\geq 24 \text{ h}$)
- UV exposure – by Laser/LED Direct Writing or with a greyscale mask in a mask aligner
- Aqueous-alkaline development

Subsequently, the obtained resist master is typically transferred into the substrate by an etch processes or replicated into other permanent materials using metallization and electroplating or UV-moulding.

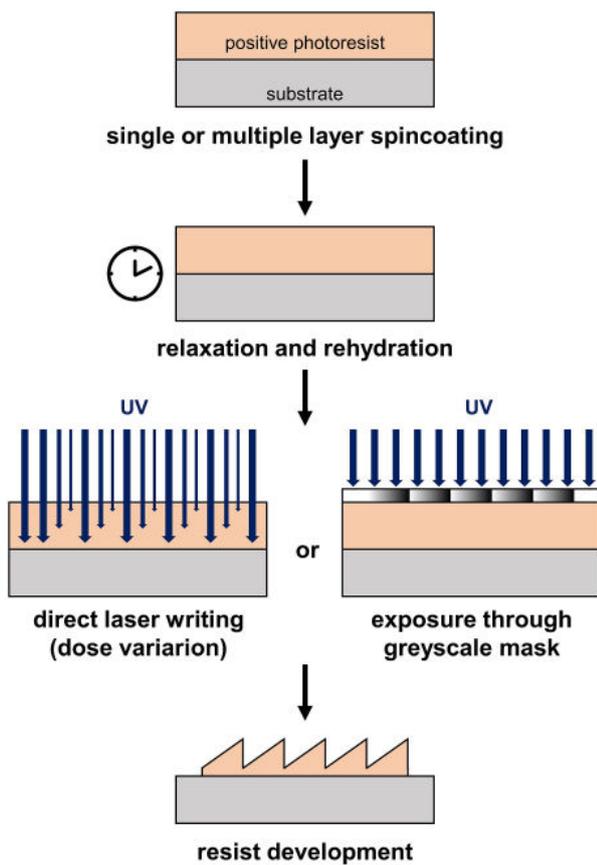


Fig. 2. Greyscale lithography process using a positive greyscale resist and different options for UV exposure: direct laser writing (left) and mask aligner exposure using a greyscale mask (right).

Chemical background of greyscale lithography with positive photoresists

Greyscale resists described here are positive photoresists based on novolak as polymeric binder and diazonaphthoquinones (DNQ) as photoactive compound (PAC) (Figure 3), dissolved in organic solvents. DNQ acts as a dissolution inhibitor for the novolak in aqueous-alkaline developers.

Upon UV exposure of the resist, photolysis of DNQ under release of nitrogen sets in and a ketene intermediate is formed. The latter reacts with water, which is absorbed into the resist from ambient air, forming indene carboxylic acid (ICA). Importantly, ICA acts as dissolution promoter increasing the dissolution rate of the novolak matrix in aqueous-alkaline developers. While the unexposed DNQ/novolak resist has a very small, but non-zero dissolution rate leading to minor dark erosion during development, after exposure the dissolution rate increases by several orders of magnitude for the ICA/novolak system. More detailed information on the chemistry of DNQ/novolak resists is compiled in [1].

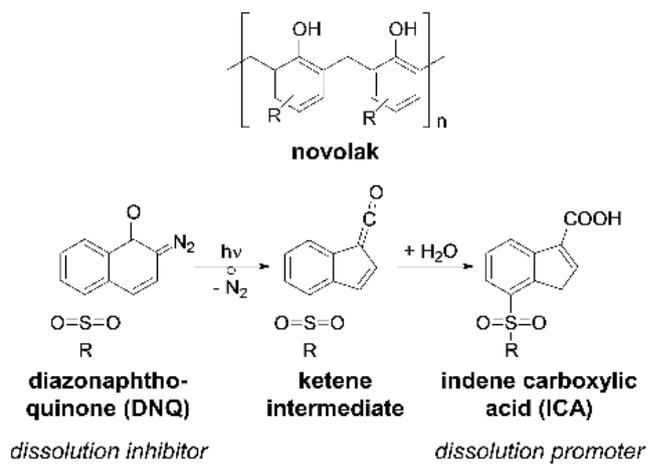


Fig. 3. Structure of novolak used as polymeric binder in positive photoresists (top) and photoreaction of DNQ photoactive compound during exposure of the resist (bottom).

In standard binary UV lithography the solubility change due to exposure is supposed to be as high as possible – leaving insoluble resist in unexposed areas and completely soluble resist in exposed areas. In contrast, for greyscale lithography the dissolution rate of the resist should increase gradually with increasing exposure dose, which translates into a gradually decreasing resist film thickness after the development step.

In contrast to positive photoresists, greyscale patterning of a negative-tone photoresist is more complex. Negative-tone resists decrease their solubility upon UV exposure, usually due to cross-linking of the resist's polymeric binder. With exposure from above, the highest degree of cross-linking and corresponding solubility decrease would appear in the top layers of the resist film while the bottom layers would still be soluble in the developer. Hence, instable patterns would be produced, which do not adhere to the substrate.

This is why positive tone photoresists are the workhorse for greyscale lithography.

Working principles of greyscale resists

Figure 4 shows a typical response curve, i.e. dependence of the resist film thickness (FT) after development on the exposure dose, of a standard DNQ/novolak positive resist compared to the ideal response curve for a greyscale resist.

In the ideal case the response curve shows linear behavior with a constant slope, thus any dose gradient is directly transferred into a film thickness gradient. Furthermore, the curve should be steep enough to avoid excessive writing time in direct writing lithography especially for thick resist films. However, the response curve should not be as steep as that of a standard positive resist to enable precise writing of intermediate height levels.

In reality, DNQ/novolak resists do not respond linearly to increasing exposure doses. At lower doses there is extended dissolution inhibition, i.e. up to a certain dose threshold the resist is not dissolved and the film remains at its initial thickness. This is mainly due to the fact that the backbone molecules of the PACs contain more than one DNQ-group, in order to increase photosensitivity. After exposure mostly the PAC with all its DNQ moieties converted to ICA contributes to the solubility increase of the matrix, i.e. it acts as dissolution promoter, whereas incompletely converted PAC molecules hardly increase the solubility of the resist in the developer. Such completely converted PAC is the final product of consecutive reaction steps, thus leading to the nonlinear response of the resist.

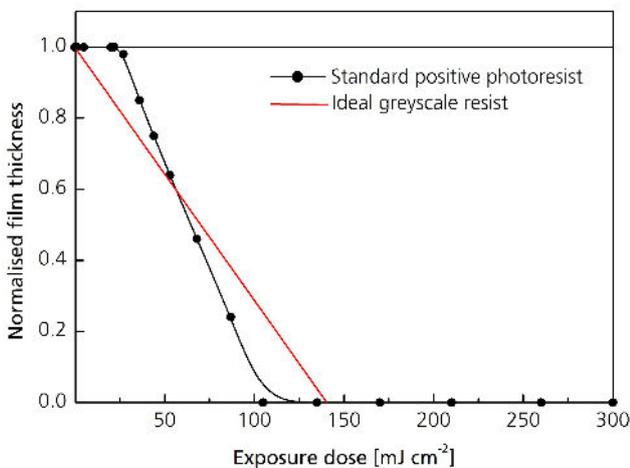


Fig. 4. Response curves (normalised film thickness FT/ FT₀ after development vs. exposure dose) of a standard binary positive photoresist and an ideal greyscale resist.

Bleaching of the resist, i.e. the decrease of its absorbance during the exposure, is another important factor influencing the resist response. Resist bleaching to a very low, wavelength-dependent residual absorbance enables the exposure of thick films in the first place. However, even the low residual absorption after complete photoreaction of the DNQ PAC attenuates the exposure light with increasing depth in the resist film.

In thin resist layers with film thickness <5 μm, the penetration depth of the exposure light is higher than the film thickness (Figure 5, left). As a result, the PAC conversion is only dependent on the local exposure dose leading to a lateral variation of the dissolution rate of the resist film in the developer. In contrast, for thick resist films the penetration depth of the exposure light is smaller than the film thickness (Figure 5, right). Thus, the rate of resist bleaching contributes to the variation of PAC conversion at different doses and at different depths of the layer. As a result, the dissolution rate of the resist also varies in z-direction, which adds to the film thickness gradient after development.

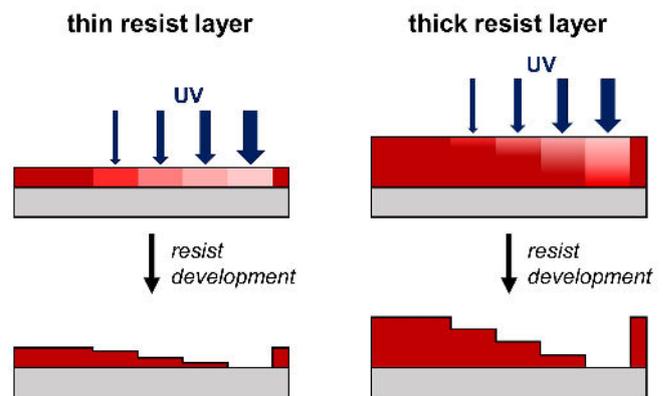


Fig. 5. Greyscale exposure of thin (FT ≤ 5 μm) and thick (FT > 5 μm) resist films.

Particularly for thick resists films, which require higher exposure doses for greyscale structuring down to the bottom, the release of nitrogen associated with the conversion of DNQ-moieties can become an issue. Due to the release of too much N₂ at once gas bubbles can form especially in the lower parts of a thick resist layer in areas with high exposure dose. Similarly, exposure using high-intensity light (e.g. laser or high power LED) can lead to a high local rate of nitrogen release, resulting in bubble formation. The bubbles remain in the exposed resist and become visible as surface defects after development of the structure.

Consequently, the maximum film thicknesses and exposure dose are limited by the outgassing behavior of the resist.

Generally, greyscale lithography has a much smaller process window than standard binary lithography, i.e. processing conditions have a higher impact on the patterning results. This includes e.g. softbake conditions, relaxation times before exposure for rehydration, exposure wavelength and intensity, developer chemistry and concentration, or ambient conditions in general. The overall process is also much more sensitive to any variation in photoresist composition compared to binary lithography, particularly requiring for high batch-to-batch reproducibility in resist manufacturing.

How to design a greyscale resist

The requirements for a good greyscale lithography resist are the following:

- Good film forming properties
- Linear response and reduction of contrast in comparison to binary resist, negligible initial solubility inhibition
- Sufficiently steep response curve to avoid long exposure, but not too steep to allow greyscale response
- Efficient bleaching of photoactive compound, i.e. low residual absorbance
- No excessive outgassing during exposure
- High batch-to-batch reproducibility
- Sufficient stability & chemical compatibility for pattern transfer procedures

Generally, finding the best recipe for a greyscale resist means finding a suitable trade-off between all, partly contradicting effects of PAC structure and novolak matrix. Considering the PAC, the number of DNQ groups attached to the backbone, the molecular structure of the backbone as well as the connectivity at the DNQ moiety (DNQ-5-SO₃ ester vs. DNQ-4-SO₃ ester) strongly influences its UV-absorption and photosensitivity. Furthermore, the dissolution inhibiting and promoting effects on the novolak matrix strongly depend on the number and configuration of DNQ groups at the PAC-backbone. Miscibility of the PAC with the matrix polymer as well as its stability and aging behaviour has to be considered.

By varying the novolak matrix, in particular the relative content of p-, m- and o-cresol moieties and its molecular weight, the dissolution behaviour in different aqueous-alkaline developers can be tailored.

Furthermore, miscibility with the chosen PAC and solvent as well as film forming properties of the resist should be carefully optimized.

GREYSCALE RESISTS ma-P 1200G

With the **ma-P 1200G series**, which is a result of our product development aiming to fulfil the aforementioned requirements, micro resist technology GmbH is able to offer an efficient greyscale resist suited for patterning thin as well as thick resist layers.

The series comprises three standard products, ma-P 1215G, ma-P 1225G, and ma-P 1275G, as well as an experimental product, ma-P 1295G_XP. All resists have the same solids composition but vary in their solids contents, thus covering different film thickness ranges under standard processing conditions. Experimental products with alternative compositions are under development.

Film thickness

The three standard resists of the series allow the preparation of 1 µm to 60 µm thick films in one spin-coating step. Available film thicknesses under standard spin-coating conditions (1000-6000 rpm, 30 s) are shown in Figure 6. To achieve 60 µm film thickness with ma-P 1275G in one step, a medium spin-speed is applied for a very short time (1000 rpm, 4 s). The resulting film is smooth and shows good homogeneity with a total thickness variation of 4% (Figure 7).

Thicker layers of up to 100 µm can be obtained using ma-P 1275G in a double coating process (2x 1500 rpm, 5 s) with a short intermediate bake (80 °C for 5 min). A temperature ramp with several halting times is beneficial for the softbake of ≥ 40 µm thick films.

Before exposure it is crucial that enough water has been re-absorbed into the resist film to enable the desired photoreaction to the indene carboxylic acid. Hence, thicker resist films (FT > 15 µm) require time for re-hydration between softbake and exposure. This time increases exponentially with the film thickness: a 30 µm thick film requires approximately 1 h for re-hydration, for 60 µm we recommend 12-24 h. [2]

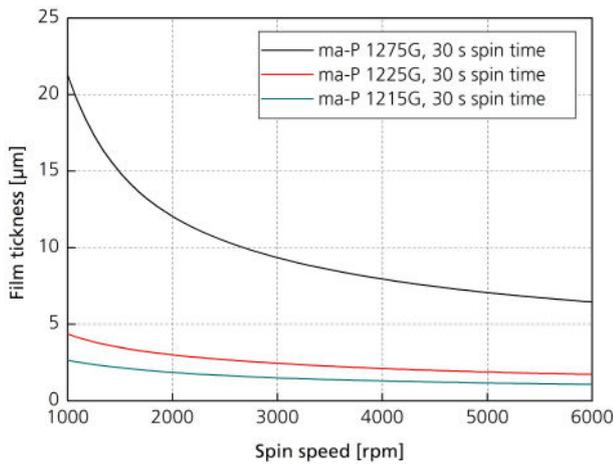


Fig. 6. Film thickness obtained with different resists of the ma-P 1200G series by spin coating with 30 s spin time.

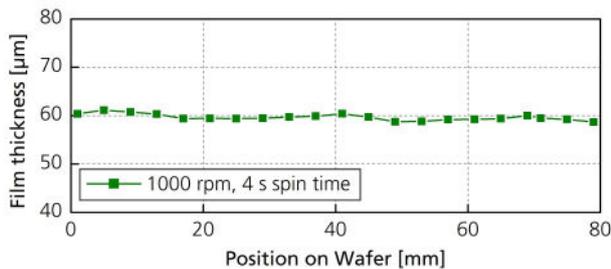


Fig. 7. Top: photograph of a 4" Si wafer coated with a 60 μm thick ma-P 1275G film after spin-coating (1000 rpm, 4 s spin time) and edge bead removal. Bottom: Measured film thickness across the wafer after the softbake.

Reduced outgassing

The composition of ma-P 1200G resists has been adjusted to reduce bubble formation due to outgassing during high dose or high intensity exposure. This makes this resist suitable for laser direct writing and for the greyscale fabrication of deep structures. As an illustration, Figure 8 compares nitrogen bubble formation in ~60 μm thick films of standard, binary photoresist ma-P 1275HV and greyscale resist ma-P 1275G after high-intensity LED exposure. For the binary resist extensive bubble formation is observed in the undeveloped resist layer already at an exposure dose of 5000 mJ cm⁻². In contrast, the ma-P 1275G greyscale resist does not show any such defects after 5000 mJ cm⁻² exposure. Only at the fourfold exposure dose a slight bubble formation sets in.

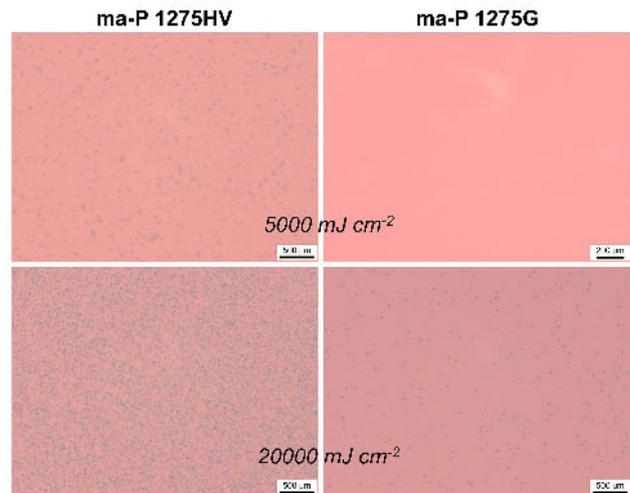


Fig. 8. Optical microscope image of bubble formation due to resist outgassing in ~ 60 μm thick films of ma-P 1275HV (left) and ma-P 1275G (right) after LED exposure at 410 nm (I = 275 mW cm⁻²). Exposure dose: 5000 mJ cm⁻² (upper row) and 20000 mJ cm⁻² (bottom row).

Greyscale exposure

The ma-P 1200G resist is effective for exposure at wavelengths of 330 - 450 nm. The bleaching of the resist during exposure due to the photolysis of the PAC, being essential for the efficient exposure of thick films, can be observed in absorption spectra of unexposed and exposed films on quartz substrates (Figure 9). Notably, the residual absorption after exposure is wavelength-dependent. It attenuates the exposure light with increasing depth in the resist film. The intensity reduction can be calculated using Lambert-Beer's law, and is stronger for lower exposure wavelengths (Figure 9). Hence, in order to pattern thick resist films, exposure using monochromatic laser or LED light of higher wavelengths, within the sensitivity range of the photoresist, is necessary. For medium film thickness (10-20 μm) exposure at wavelengths >350 nm and for film thickness >20 μm exposure at wavelengths ≥400 nm is beneficial.

As outlined above, a reduced contrast compared to binary lithography is a requirement for greyscale resists. Figure 10 compares the contrast curves of a 5 μm thick film of ma-P 1200G and a 4 μm thick film of ma-P 1200, the latter being a resist for standard binary UV lithography. Contrast values [γ] of 1.1 for the greyscale resist and 1.6 for the binary resist were measured under the given processing conditions. In addition to the reduced contrast, ma-P 1200G greyscale resists show significantly less initial dissolution inhibition, i.e. already at low exposure doses a gradual decrease of the film thickness is observed.

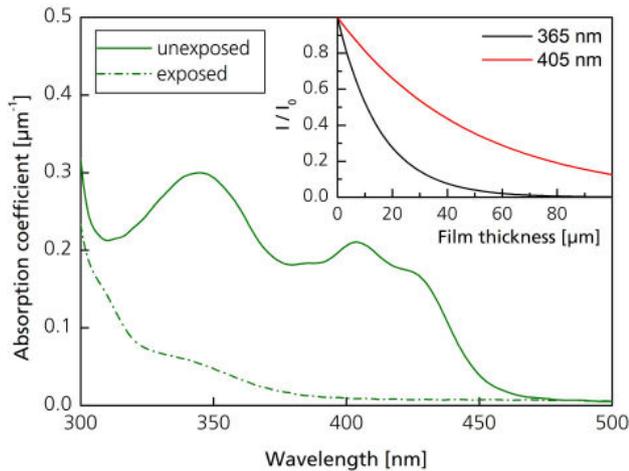


Fig. 9. UV/vis absorption spectra of an unexposed and exposed ma-P 1200G resist film, measured on a 1 x 2.5 cm² quartz substrate in 4 μm film thickness (absorbance normalised to 1 μm film thickness). Inset: Resulting light attenuation at 365 nm and 405 nm calculated using Lambert-Beer's law.

Greyscale structuring with low contrast can also be achieved in thicker or very thin films of ma-P 1200G. The contrast value γ for a 28 μm thick film of ma-P 1275G was found to be 0.9 (Figure 10). Figure 11 shows a greyscale response to graduated dose increase in 2.5 μm thick layers of ma-P 1200G. This example also shows that the influence of the developer on the resist response is crucial. In case of a too low developer concentration, developing time is increased and unwanted initial dissolution inhibition is observed. This is just one example for the influence of the processing conditions on the patterning result, i.e. even for greyscale lithography is smaller than in binary lithography.

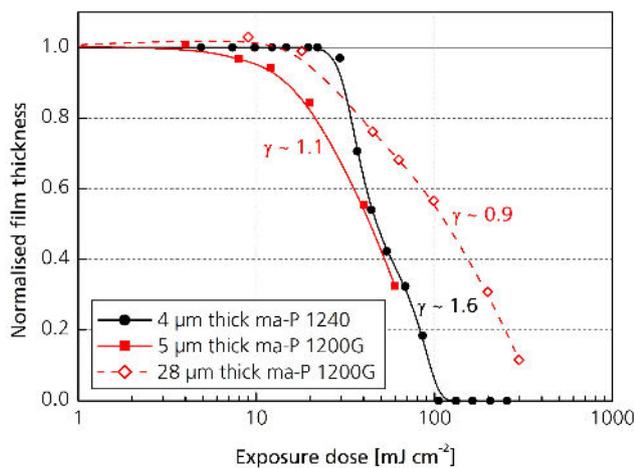


Fig. 10. Contrast curves of 5 μm and 28 μm thick ma-P 1200G and of 4 μm thick standard positive photoresist ma-P 1200 after mask aligner broadband exposure and development in 0.26N TMAH (ma-P 1200G) and 0.15N NaOH (ma-P 1200), respectively.

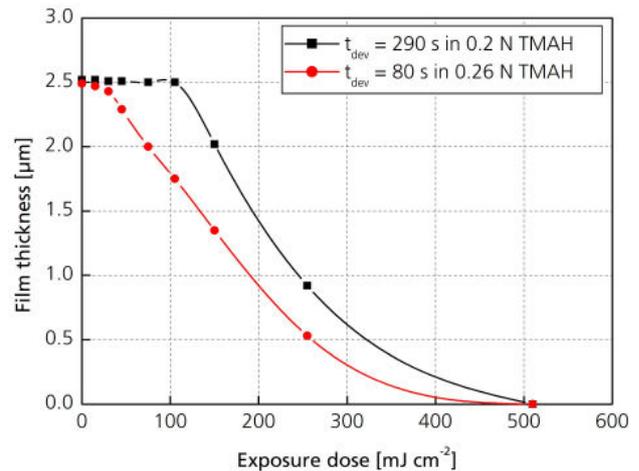


Fig. 11. Response curves of 2.5 μm thick ma-P 1225G with monochromatic 365 nm LED exposure ($I = 150 \text{ mW cm}^{-2}$), development in 0.2N and 0.26N TMAH.

Development

TMAH based developers are recommended for ma-P 1200G resists, mr-D 526/S for ma-P 1215G and ma-P 1225G, and ma-D 532/S with a higher TMAH concentration for the thicker resist films obtained with ma-P 1275G and ma-P 1295G_XP. TMAH ensures a lower surface roughness than NaOH or KOH based developers. This is due to the fact that small molecules such as NaOH have a tendency to intrude easier into channels in the novolak matrix formed by intramolecular hydrogen bonds. This can cause holes during development of incompletely exposed resist areas, noticed as higher surface roughness. The much bigger TMAH molecule reduces this risk considerably.

A too long developing time due to a low developer concentration (as in Figure 11) will cause surface erosion, too, even with TMAH. This is why micro resist technology GmbH offers ready-to-use developers adjusted to the different resists.

Patterning examples

The ma-P 1200G resist series is optimized to achieve smooth greyscale patterning in thick resist films of 50-60 μm. Figure 12a shows an example of greyscale patterning using an LED direct writing tool (Tabletop Mask Aligner μPG501 by Heidelberg Instruments) at 390 nm exposure wavelength. A pyramid hole was patterned into a 58 μm thick ma-P 1275G film.

The greyscale structures in Figure 12b were fabricated by mask aligner exposure using a greyscale mask, technology and design by Benchmark Technologies: a mask with optical density gradients e.g. for saw tooth patterns

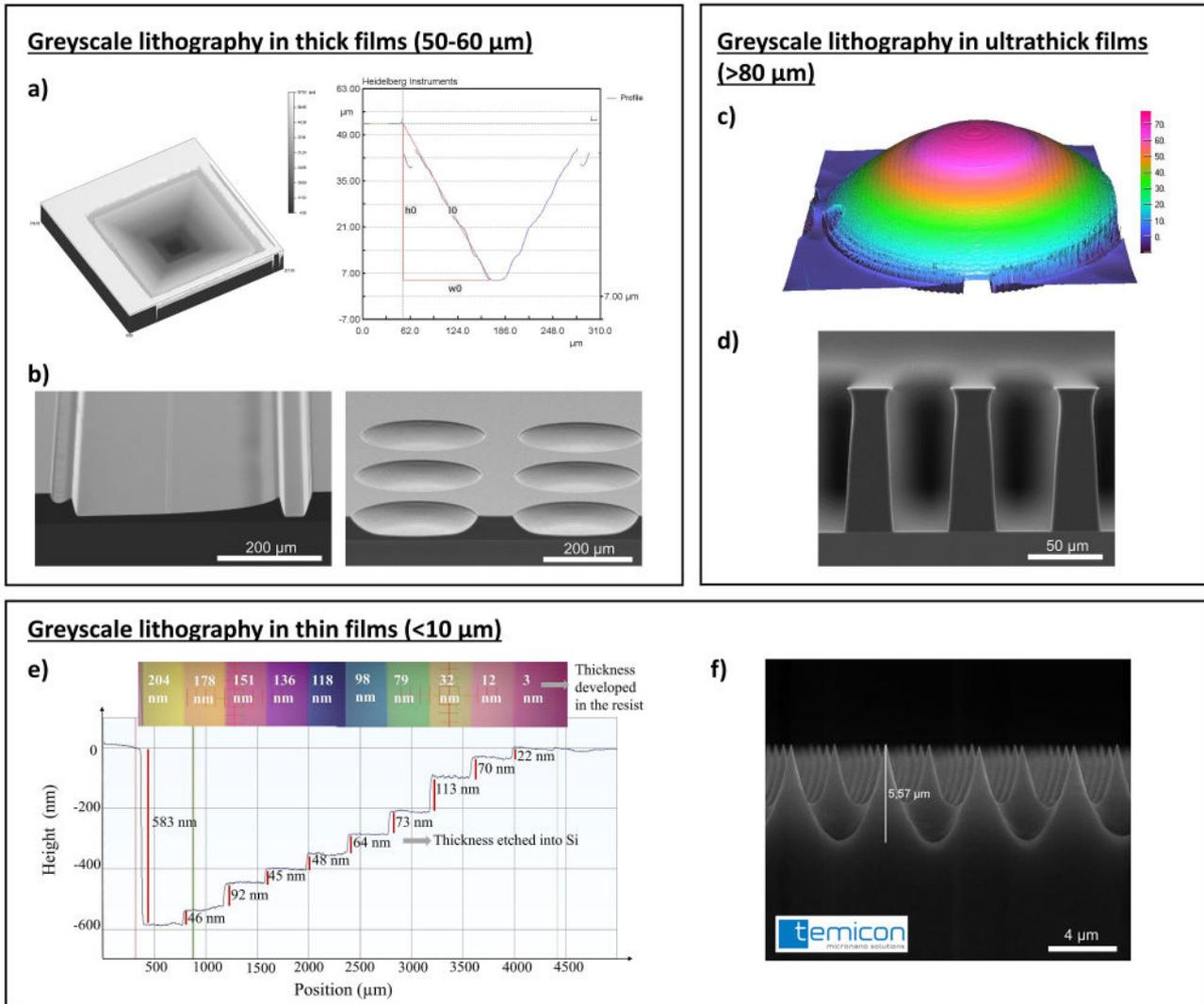


Fig. 12. Examples for greyscale patterning using ma-P 1200G series: a) Confocal microscope image of 53 μm deep pyramid hole in 58 μm thick ma-P 1275G (left) and profile scan of a pyramid hole (right, $w_0 = 113.7 \mu\text{m}$, $h_0 = 47.5 \mu\text{m}$, $l_0 = 123.2 \mu\text{m}$), by courtesy of Heidelberg Instruments. b) Greyscale patterns obtained by exposure of thick films (57 μm) of ma-P 1275G through greyscale masks: slanted profile with 48 μm pattern depth (left) and 47 μm deep concave microlenses (right). Greyscale mask technology and design by Benchmark Technologies. c) Confocal microscope image of microlens (800 μm diameter, 80 μm height) obtained with direct laser writing (DWL66+, 405 nm) in 130 μm thick ma-P 1295G_XP film. Courtesy of Heidelberg Instruments. d) 100 μm thick ma-P1275G after standard binary UV lithography with mask aligner broadband exposure. e) Heights [nm] and optical microscopy image of 10 strips patterned in 500 nm thick film of diluted ma-P 1225G resist and the corresponding profile etched into Si (plasma etching with Cl_2 , He/O_2 , HBr). [3] f) 5.6 μm deep moth eyes patterns in 10 μm thick ma-P 1275G film as master for electroplating, manufactured by laser interference lithography at 355 nm. Courtesy of temicon GmbH.

and concave lens patterns. The patterns shown were obtained in a 57 μm thick film of ma-P 1275G. During process development it proved beneficial to apply a post development bake at 90 - 95 $^\circ\text{C}$ in an oven using a temperature ramp. This bake did not result in a pattern reflow, but smoothed the resist surface avoiding unwanted pattern roughness due to pixels in the mask design.

In order to enable the preparation of ultrathick resist layers (>80 μm) we adapted the basic resist recipe by increasing the solids content, giving the prototype resist ma-P 1295G_XP. Using this resist up to

80 μm deep greyscale structures were fabricated using a Heidelberg Instruments DWL66+ direct write lithography system with a 405 nm laser (Figure 12c). BEAMER software by GenISys was applied to correct the direct writing process compensating lateral development effects. Trying even deeper greyscale patterning is limited due to N_2 bubbles trapped in the lowest parts of the resist film. An ongoing material design R&D project aims at stretching this limit to enable greyscale patterns of 100 μm or even deeper in good quality.

Binary exposure of ma-P 1275G in standard

UV lithography with mask aligner broadband exposure demonstrates the potential of the resist material. Patterning 100 µm thick resist (obtained by double coating) was possible with an aspect ratio of 3:1 (Figure 12d). Most DNQ/novolak based positive photoresists, which are designed for standard binary lithography, show significant limitations regarding their patterning in 100 µm thick films.

Besides thick and ultrathick films, the ma-P 1200G resist series also allows greyscale patterning of very thin films. Figure 12e shows an example of patterning a 500 nm thin film, which was fabricated by diluting ma-P 1225G resist with solvent. [3] Processing conditions were optimized, including a pronounced softbake, in order to flatten the contrast curve. By stepwise exposure with a tabletop micro pattern generator (µPG101, Heidelberg Instruments) at 405 nm, staircase structures with 20 nm step height were obtained. These were transferred into the Si substrate by dry etching resulting in 50 nm step height of the final structure. Figure 12f exemplifies greyscale structuring of a 10 µm thick film of ma-P 1275G using laser interference lithography, creating a continuous, greyscale-like intensity distribution of the exposure light. While with standard binary photoresists having highly non-linear response curves structures with steep sidewalls would be generated, in the example using ma-P 1200G the intensity distribution is directly transferred into graded conic structures with a height of 5.6 µm. These structures serve as template for creating moth eye patterns.

Pattern transfer

Generally, the 2.5D pattern obtained by greyscale lithographic structuring are used as resist master and can be transferred by the following processes

- dry or wet chemical etching into the substrate
- replication by electroplating
- replication by UV-moulding

As a proof-of-concept we replicated a concave microlens array (MLA), which was fabricated by direct laser writing (DLW) using ma-P 1275G as greyscale resist, by UV-moulding (Figure 13). The greyscale resist on a 4" silicon wafer was patterned in a Heidelberg Instruments DWL66+ direct writing tool with a 405 nm laser. The original film thickness was 40 µm, while the depth of the lens structures is ~20 µm with a lens width of ~30 µm. In a first step, the master was copied into its negative image, i.e. convex lenses, generating a working stamp for further replication steps. We used OrmoStamp® as UV-curable hybrid polymer, a material dedicated to the fabrication of working stamps for UV moulding and Nanoimprint Lithography. [4] An appropriate amount of OrmoStamp® resin was dispensed directly onto the greyscale resist master. A glass substrate, pre-treated with OrmoPrime08 as adhesion promoter, was carefully laid down onto the structure. The stack was shortly annealed at 50°C on a hot-plate. Then, the stack was exposed to UV-light in a MA6 mask aligner resulting in fast curing of the OrmoStamp® layer. Manual separation of the working stamp from the

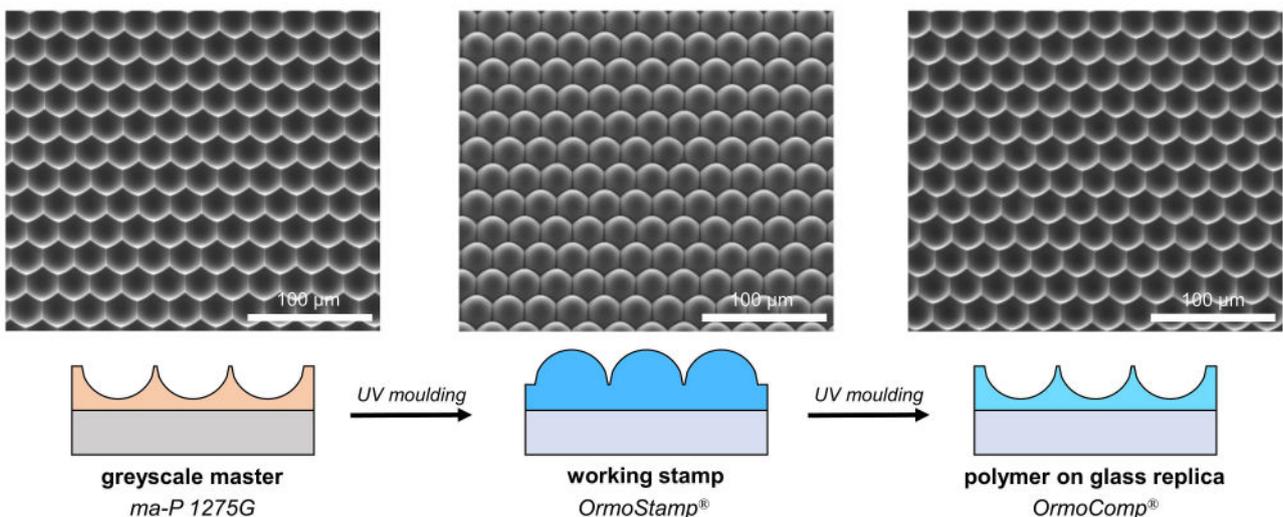


Fig. 13. Pattern transfer of greyscale structures in ma-P 1275G, concave lenses, by UV moulding with OrmoStamp® to form a working stamp and subsequently with OrmoComp® to form final concave lens structures.

master was performed and the working stamp was rinsed with acetone to remove any remaining contamination by the positive resist. After a post exposure bake on a hot-plate (130 °C, 30 min), an antisticking layer was applied to the working stamp by chemical vapour deposition.

With the thus obtained working stamp multiple polymer-on-glass replicas of the original structure can be fabricated. For this purpose, we utilized the hybrid polymer OrmoComp®, a material dedicated to the fabrication of enduring microoptical structures with high precision and long-term reliability. [5] The OrmoComp® replica was fabricated analogous to the first UV-moulding step, including dispensing of the uncured resin directly on the MLA structure, deposition of the substrate, curing with UV-light, and clean separation of the working stamp from the replica. The obtained concave MLA structure is a copy of the DLW-fabricated resist master with the original pattern polarity (Figure 13).

CONCLUSION

ma-P 1200G is a photoresist series particularly designed for greyscale lithography, matching the special requirements of that patterning method in both laser direct writing and greyscale mask exposure processes. The resist is able to cover various application scenarios, in particular greyscale structuring with high vertical resolution of very thin (500 nm) and thin (< 10 µm) films to very thick (up to 80 µm) films. Essential for its performance are its reduced initial dissolution inhibition at low exposure doses leading to a reduced contrast and an extended linear part of the response curve compared to standard DNQ/novolac resists. Furthermore, the resists show efficient photobleaching and reduced outgassing, enabling the greyscale structuring of thick resist layers using high light intensities and exposure doses.

Applications of the resists are not restricted to greyscale lithography, though. Additionally, other UV lithography processes can be applied to successfully pattern the resists. This includes binary standard lithography of 100 µm thick films with an aspect ratio of 3:1 on the one hand, and 20 nm vertical pattern resolution in a 500 nm thin film achieved with a direct writing process on the other hand. The maximum pattern depth in greyscale lithography is 80 µm for the existing resist composition. Further R&D activities aim at even higher greyscale pattern depths beyond 100 µm.

After the generation of a 2.5D structure by greyscale lithography, it typically serves as a resist master for sub-

sequent pattern transfer. We demonstrated a UV-moulding process using a 4" resist master, which was fabricated by direct laser writing in ma-P 1200G as greyscale resist. The obtained concave microlens array structure was transferred into a working stamp using the hybrid polymer material OrmoStamp® and subsequently replicated into OrmoComp® generating polymer-on-glass concave microlens arrays for permanent applications.

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