OPTIMAL

Automated Maskless Laser Lithography Platform for First Time Right Mixed Scale Patterning

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Lithographic patterning properties

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1. Executive Summary

This document represents the Deliverable D1.2 "Lithographic patterning properties" of the OPTIMAL Horizon Europe funded project. It describes the properties of lithographic patterning for a positive & a negative tone resist series for the generation of binary (2D), greyscale 2.5D and 3D patterns by using 1) a mask based/ aligner process, 2) one photon & a two/ multi photon laser lithography process as well as a 3) laser interference lithography process. The document also includes the summary of the chemical, optical, mechanical, and thermal properties of the generated resist patterns.

As reported in Chapter 2, this deliverable is the result of the activities planned in the Task 1.2 "Lithographic patterning of positive and negative tone resist formulations and characterization of the generated patterns" of the OPTIMAL Description of the Action document.

The lithographic patterns and their characterization obtained using positive and negative tone resist are described in Chapter 3. The properties of the generated patterns using mask-based exposure is reported in Chapter 3.1. The properties of the lithographic patterning using one-photon laser lithography, laser interference lithography and two-photon lithography are described respectively in Chapter 3.2, Chapter 3.3, and Chapter 3.4.

Chapter 4 is focused on the chemical, optical, mechanical, and thermal properties of the generated resist patterns.

Finally, the key results are reported in Chapter 5. Positive resist layers up to (190 – 200) µm can be generated by the use of the newly developed mr-P 22G_XP 2nd Gen prototype resist formulation as targeted within OPTIMAL project. With the current mr-P 22G_XP resist formulation high precision 2.5D greyscale structures and exposure depths of up to 150 µm by one-photon lithography (1PL) and an achievable surface roughness on patterned resist surface of about 33 nm were reproducibly and defect-free obtained. With mr-DWL negative resist series up to 180 µm thick resist layers can be generated in a single coating step. Up to 500 µm thick resist patterns were successfully generated by the use of mask-based exposure using mask aligner (broadband) or UV-LED @ 405/410 nm.

2. Introduction

This deliverable is the result of the activities planned in the Task 1.2 “Lithographic patterning of positive and negative tone resist formulations and characterization of the generated patterns”. The activities performed in the Task are summarized below.

The lithographic patterning results are generated using experimental setup available at the project partners Micro Resist Technology (Germany), Joanneum Research (Austria), Slovak Centre of Scientific and Technical Information (Slovakia) and University of Zilina (Slovakia) with the aim to characterize the new developed photoresist materials and provide data for the development of the optimized virtual photomask. The lithographic patterning parameters will be used for the development of the large area substrate coating and the patterning processes, the production of masters and the validation of the OPTIMAL platform through the use cases.

Lithographic patterning process was done using the developed positive and negative resist formulations in a “medium” thickness by using 1) mask aligner exposure, 2) one photon laser direct writing at wavelength of 405 nm and 3) a two or multi photon absorption process by the use of a fs laser at a wavelength of 780 nm and 4) Interference lithography by the use of a laser at a wavelength of 405 nm.

The lithographic patterning was done in the full range of the target resist thickness (sub-µm to sub-mm) by selecting at least two representative thicknesses from the targeted thickness range.

Characterization of generated resist patterns are concerning thickness/depth, sensitivity, absorption before/after exposure, resolution, roughness, bubble formation, development time, etc. Characterization of chemical, thermal, mechanical, and optical properties of generated resist patterns is also done.

3. Lithographic patterning of positive & negative tone resist formulations and characterization of the generated patterns

3.1. Lithographic patterning in mask-based exposure

Positive resist: Using a new positive resist formulation (code: mr-P 22G_XP), resist patterns for different thicknesses were obtained by broadband mask aligner (MA/BB) lithography, as well by LED @ 410 nm with a grey scale mask exposure (Figure 1 - Figure 3). The used High Energy Beam Sensitive (HEBS) glass masks (by Canyon Materials Inc.) are capable of more than 500 grey levels. The minimum width of a grey level is 0.1µm. The HEBS glass mask applied here did not cover the full transmission range from 0% to 100%, leading, that the resulting pattern height was lower than the original film thickness.

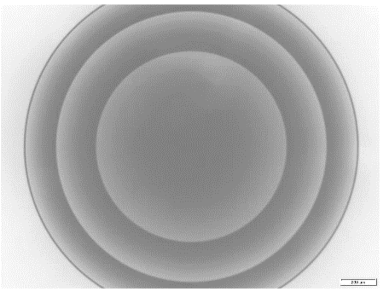


Figure 1. 2 mm diameter Fresnel lens pattern in the HEBS glass mask; dark areas not completely dark

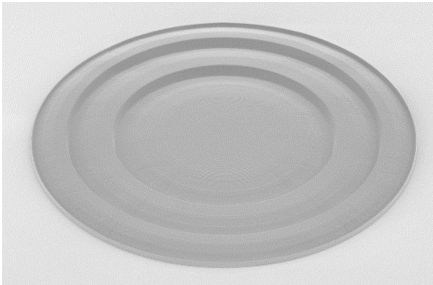


Figure 2. 60 µm high mr-P 22G_XP Fresnel lens, original film thickness ~ 100 µm

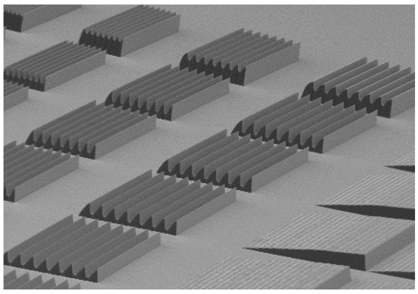


Figure 3 max. 70 µm high mr-P 22G_XP saw tooth patterns in different heights and pitches, original film thickness 96 µm

Negative resist: Using a commercially available negative resist (code: mr-DWL), resist patterns for different thicknesses beyond 100 µm were obtained by broadband mask aligner (SUSS MA/BB@ 405 nm) lithography, as well by LED @ 410 nm exposure as shown in Figure 4 (a. – d.).

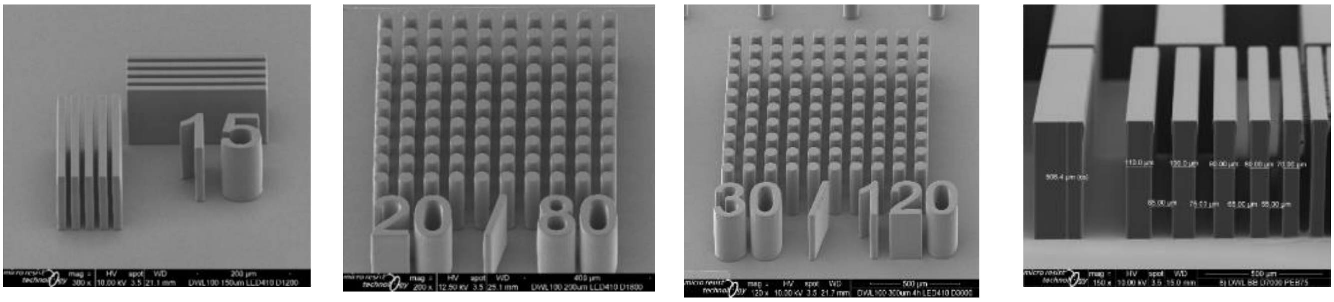


Figure 4 a. Film thickness (FT): 150 µm, resolution: 15 µm, aspect ratio (AR): 10; b. FT: 200 µm, Resolution: 20 µm, AR: 10; c. FT: 300 µm, Resolution: 30 µm, AR: 10; d. FT: 500 µm, AR: ~ 8

3.2. Lithographic patterning by one-photon laser lithography (1PL)

One-photon lithography with a laser wavelength of 405 nm was used for direct laser writing with the aim to obtain defined resist pattern profiles in thick films of positive and negative resist layers. For three of the OPTIMAL use cases greyscale 2.5D pattern by the use of a positive resist are targeted to obtain. For one of the OPTIMAL use case up to 500 µm thick patterns are targeted to obtain by the use of a negative resist.

Positive resist: The positive tone resist mr-P 22G_XP provides a large modulation depth window. To demonstrate it, a relationship between laser power and exposure depth after development was established and a triangular shaped profile with a linearly inclined surface was patterned on a thick layer of mr-P 22G_XP resist layer. The measurement by a stylus profilometer of the obtained resist pattern profile is shown in Figure 5. The laser power range applied allow a structure/pattern formation with a depth range of 110 µm starting from 35 µm to 145 µm. The patterning range can be expanded depending on the available laser power range.

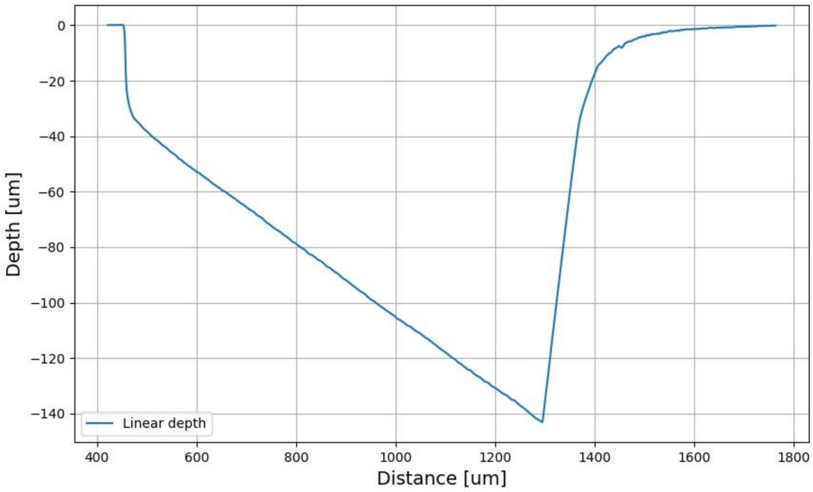


Figure 5. Stylus profilometer measurement of the linearly inclined, greyscale pattern in thick mr-P 22G_XP resist layer

The analysis of applied modulation depth by patterning of grooves of symmetric triangular profiles is shown in Figure 6. Three arrays of 11 grooves with increased pattern depth from app. 10 µm to 90 µm were obtained by gradually increased power to increase the penetration depth.

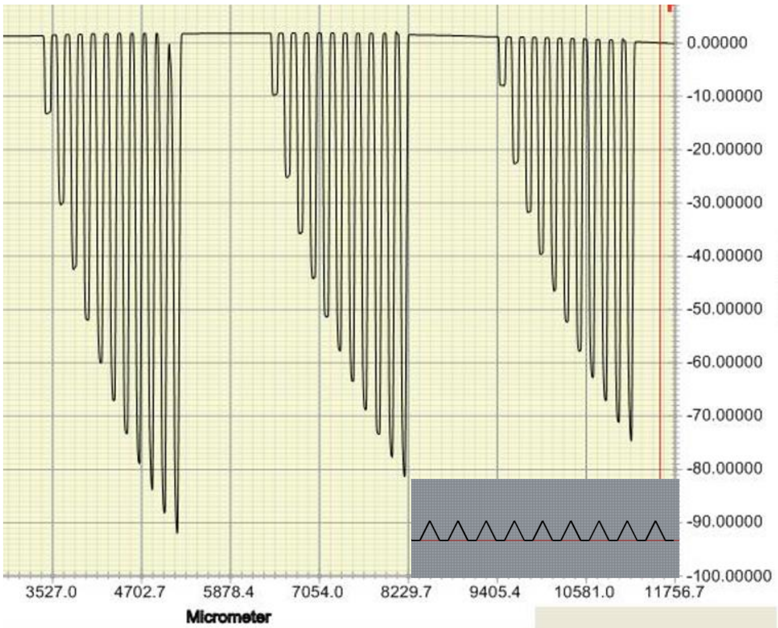


Figure 6 Stylus profilometer measurement on set of profiling with increasing the depth patterned in mr-P 22G_XP resist layer.

With the current mr-P 22G_XP resist formulation high precision 2.5D structures and exposure depths of about 150 µm by one-photon lithography were reproducibly and defect-free obtained

The following experiment focused on the quality, respective roughness of the patterned resist surface. Utilizing techniques to smoothen the exposed resist surface, such as repeated exposure, tighter patterned line spacing and defocusing by increasing the distance of the sample from the objective, an average line roughness per 50 µm segment of R_a = 33 nm was achieved. The objective used had a 4x magnification with NA of 0.1 and optimizing the optics could further smoothen the patterned resist surface. Figure 7 shows the differential interference contrast (DIC) microscopy (a), and the profilometer measurement of patterned rectangular grooves with declared roughness (b).

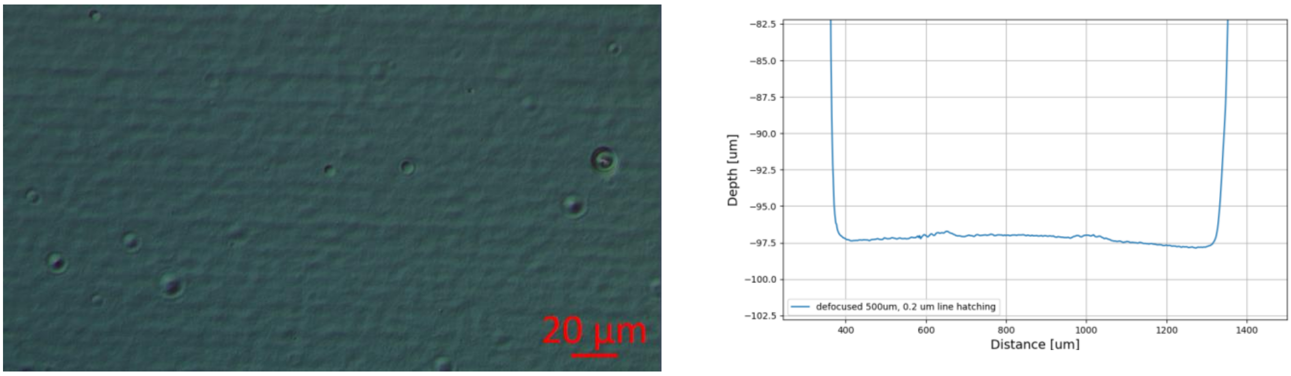


Figure 7 a. DIC microscopy image of the surface of the exposed positive resist; 7b. Stylus profilometer measurement of line roughness perpendicular to the writing direction.

With the available exposure depth modulation of up to 150 µm and a surface roughness of patterned resist of about 33 nm, mr-P 22G_XP is well suitable for the purposes of the OPTIMAL project.

Negative resist: Figure 8 shows a 1PL direct writing @ 405 nm obtained using mr-DWL resist pattern having a maximum exposure height of around 130 µm.

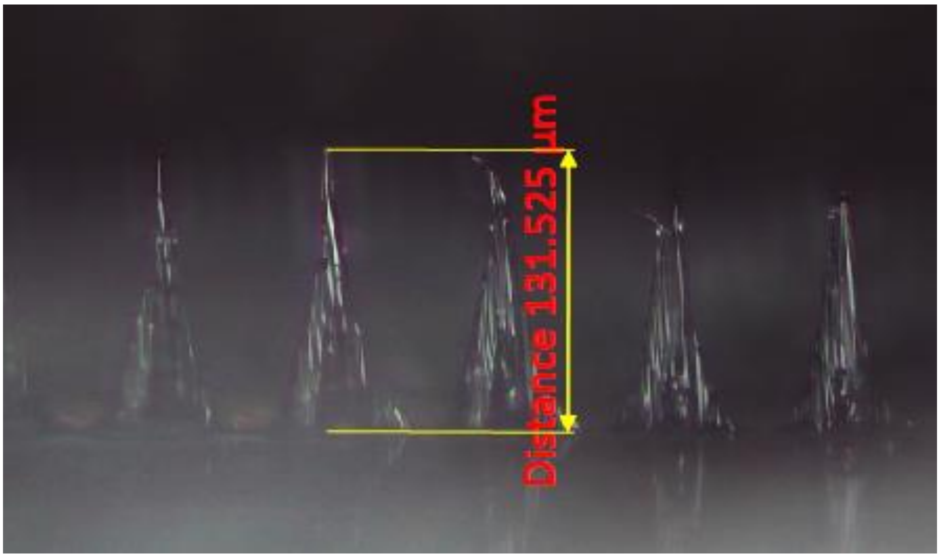


Figure 8. Optical dark field microscope image showing the maximum achieved height of a thick mr-DWL layer using one-photon lithography.

With the current mr-DWL resist formulation, resist patterns with a maximal height of up to 130 µm are obtained when laser direct writing with a single emission @ 405 nm is applied. As for the use case requiring resist patterns of up to 500 µm thickness, alternative processes are under development including the use of high power LED for exposure/patterning having a broader band width or an updated mr-DWL formulation with higher transparency.

3.3. Lithographic patterning by laser interference lithography (LIL)

In the Laser interference lithography experiments the focus was to obtain high quality gratings with sufficient exposure depth and a broad range of periods with the aim to generate hierarchical patterns. The experiments aimed to demonstrate the achievement of homogeneous structures in the area of 25 x 25 mm^2. These experiments were prepared on different positive resist thicknesses and also the grating on negative tone photoresist was tested. The quality of the gratings was examined by scanning electron microscope (SEM).

Positive resist:

Aspect ratio optimization

The exposure dose of the LIL setup was set to 220 mJ/cm^2 for all processes on mr-P 22G_XP positive tone photoresist. The application of a laser power in the exposure area of app. 2-3 mW/cm^2 leads to exposure times between 60-120 s.

In the experiments the focus was to achieve maximal exposure depth with respect to the constant period. Results obtained by adjusting of the exposure parameters and the progress of pattern depth formation on the developing time are shown in Table 1, applied on same resist thickness of 120 µm. Electron microscope pictures of cross-sections of obtained resist patterns are shown in Figure 9. The resist layers are exposed by an optical field with a period of 900 nm and were developed with different developing times. The achieved resist pattern exposure depths range from 200 nm to 1050 nm for the different applied developing times. An increasing aspect ratio (AR) with a maximal value of 1.37 at 300 s is demonstrated. Further increasing developing time caused over etching (or already attack of resist patterns) leading to an AR decrease.

Table 1. Applied LIL process parameters on thick mr-P 22_G_XP layer

mr-P 22G_XP: film thickness 120 µm, Exposure dose: 220 mJ/cm^2, Applied grating period: 900 nm							
Developing time [s]	180	195	210	225	240	300	330
Exposure depth [nm]	200	295	350	660	700	1055	790

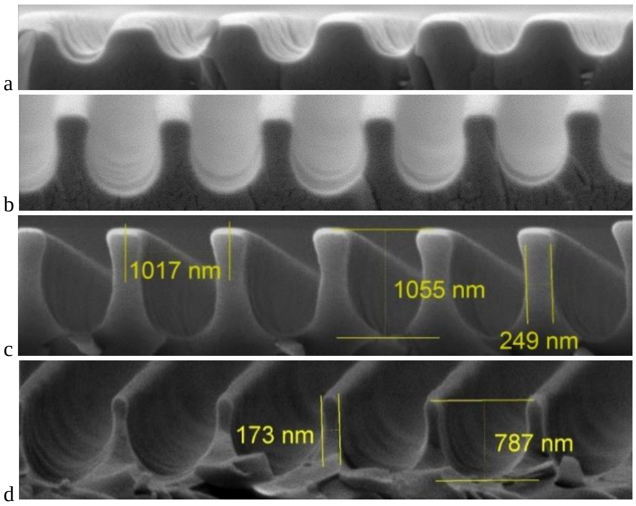


Figure 9. SEM images of exposure depth formation for different developing time a) 195 s, b) 240 s, c) 300 s and d) 350 s for grating period 900 nm

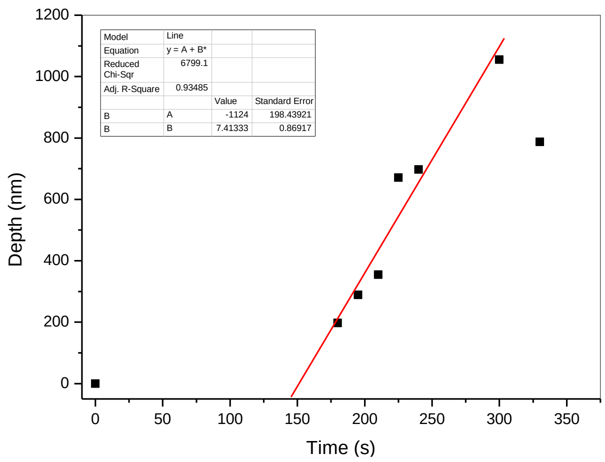


Figure 10. Depth of gratings for 900 nm period grating as a function of developing time

The graph in Figure 10 documents the relation between obtained pattern depth on the applied developing time. The formation of the pattern gratings in the surface of thick resist layers starts app. at 120 – 150 s developing time. After that time offset, the pattern depth is formed nearly linear with increasing developing time. Exceeding 300 s developing time, the unexposed surface layer is etched/attacked and the grating depth decreases. The steepness of the linear dependence documents the developing rate app. 450 nm/min.

Different grating periods

Next experiments focused on the formation of gratings of different periods with the best resolution of 250 nm up to 1 µm using the described experimental setup for laser interference lithography. The LIL setup allows very precise adjustment of the grating with an accuracy of better than 0.05 nm. It exceeds the desired resolution by an order of magnitude as standard grating adjustment better than 1 nm is not necessary. Developing times were adapted to the grating period to keep the etching/developing rate app. 450 nm/min and considering the time offset app. 120 s.

Process parameters are shown in Table 2 and SEM images of cross section of generated resist gratings with periods of 250 nm, 690 nm and 1000 nm show high aspect ratio grating quality (Figure 11).

Table 2. Applied LIL process parameters to investigate different grating periods

mr-P 22G_XP: film thickness 120 µm, Exposure dose: 220 mJ/cm^2,			
Applied grating period [nm]	1000	690	25
Developing time [s]	300	250	150

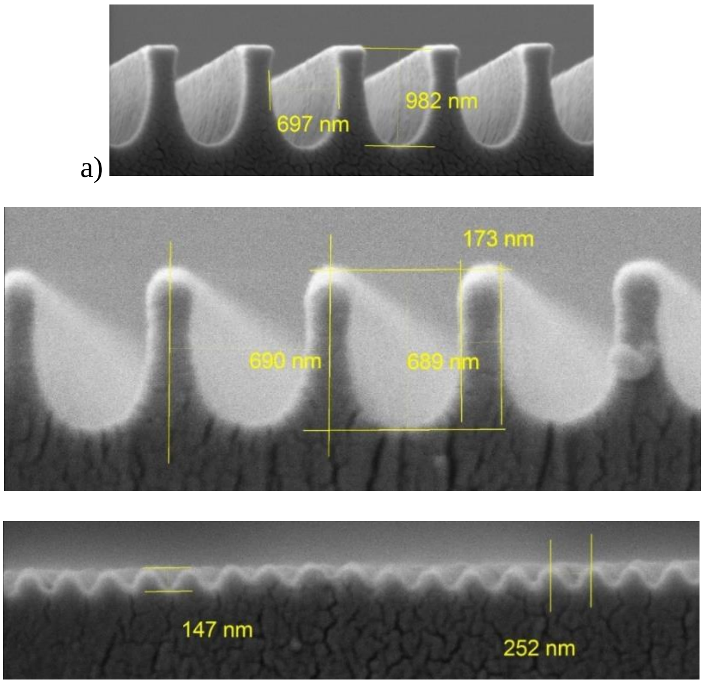


Figure 11. SEM images of generated gratings in mr-P 22G_XP resist surface with periods of a) 250 nm, b) 690 nm) c) 1000 nm

Quality of the resist gratings

Gratings were obtained on the substrates of an area 25 x 25 mm^2, demonstrating high grating homogeneity and area quality. Figure 12 documents the same quality of the grating with period of 1 µm over the resist surface. Generally, the exposed area could be much greater than 25 x 25 mm^2, however, it needs the reconfiguration of the LIL setup with greater beam expanding and prolongation of exposure times.

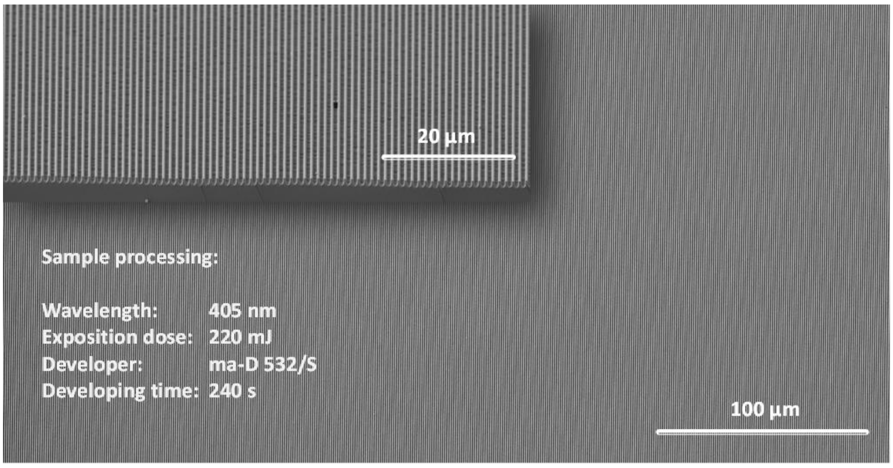


Figure 12. SEM images of the high grating quality at different places of the patterned large resist layer

Generation of 2D gratings

The LIL setup allows the formation of complicated symmetries of the grating using the accumulation of exposure dose by exposure at different sample orientation. The LIL system is capable to automatically make multiple exposures synchronized with sample rotation. This leads to patterning of highly-symmetric two-dimensional (2D) gratings. Figure 13 demonstrates the capability of the LIL to create high quality 2D gratings with a square symmetry of 1 µm period. The grating was prepared by double exposure (2 x 30 s, 2 x 110 mJ/cm^2) at 90° sample rotation between exposures. Generally, there is no limitation to create multiple symmetries by using multiple exposures. However, overall exposure dose should be 220 mJ/cm^2 including all individual exposures.

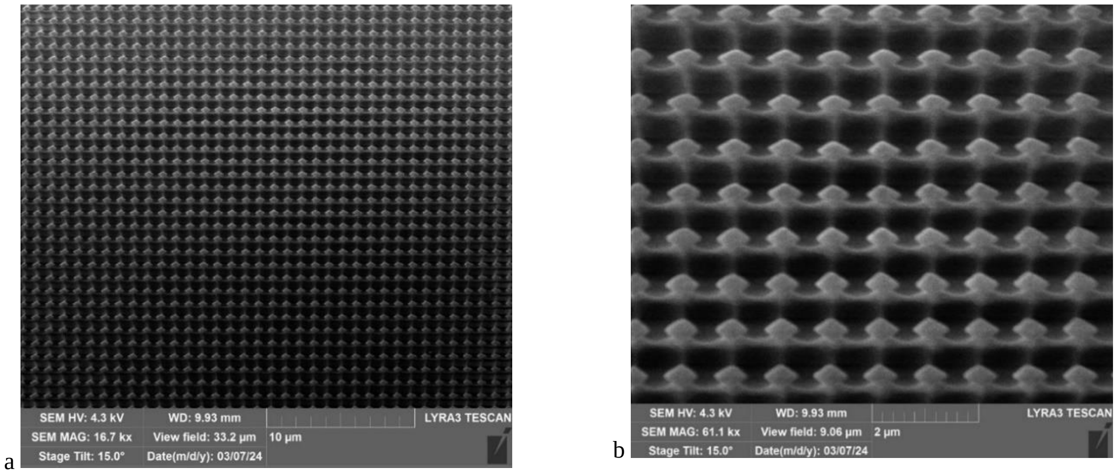


Figure 13. 2D grating patterned in thick mr-P 22G_XP resist layer: a) overall view, b) detail cross section image

Grating on thin ma-P 1200G (1st Gen) resist layers

For the formation of gratings on thinner resist layers spin coated layers of the ma-P 1200G (1st Gen) series were used: i) ma-P 1275G for 7 µm and ii) ma-P 1205G for 300 nm thin layers. Figure 14 demonstrate the grating quality on two different ma-P 1200G resist film thicknesses, showing the capability to form gratings by LIL on great scale of photoresist thicknesses.

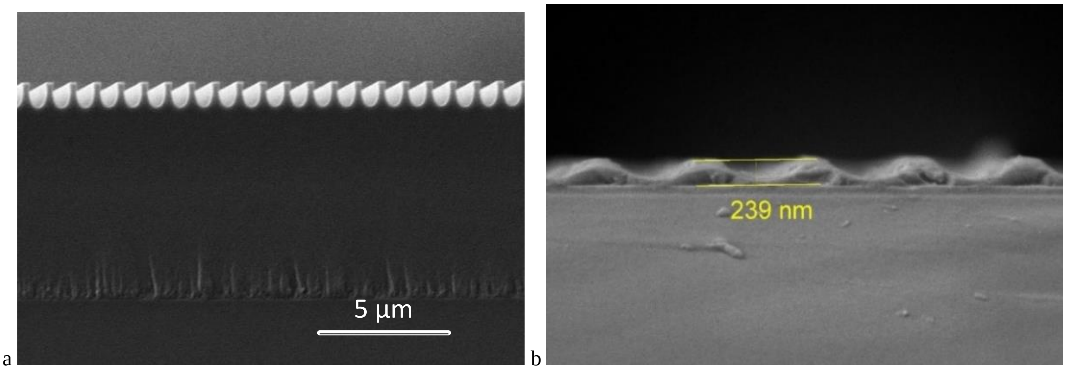


Figure 14. Gratings of 1 µm periods, patterned on ma-P 1200G resist layers surfaces of resist thicknesses of a) 7 µm and b) 300 nm

Negative resist: For the generation of LIL patterns in a negative resist, usually thin resist layers have to be used, since the exposed and crosslinked resist has to be in direct contact with the substrate surface (otherwise the resist is washed away during the developing process). Thin layers of spin coated mr-DWL negative resist with a thickness of 1 µm were used for the generation of a targeted depth of gratings of ~1 µm. The following process parameters were applied: spin coating 30 s @ 3000 rpm, prebaking 2 min @ 100°C, exposure 220 mJ/cm^2, post exposure bake 2 min @ 100°C, developing time 60 s in mr-Dev 600. Figure 15 demonstrates the achievement of a 1 µm period grating with a height of 555 nm.

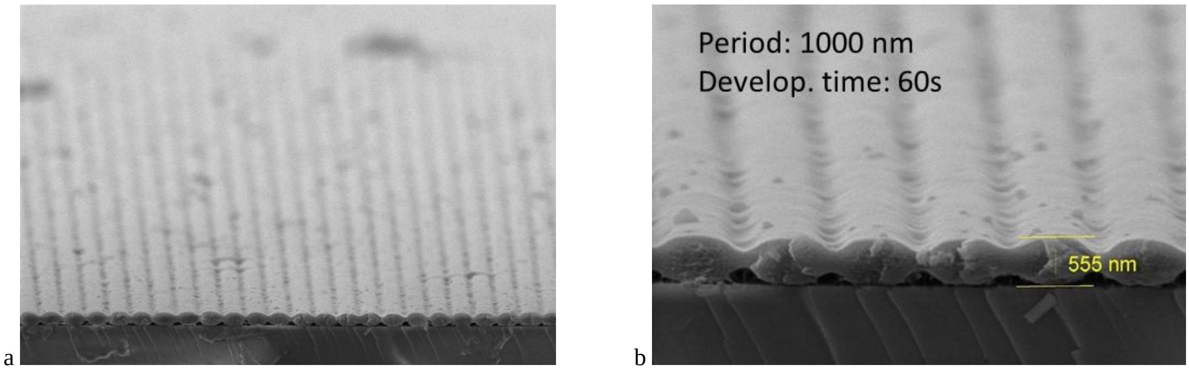
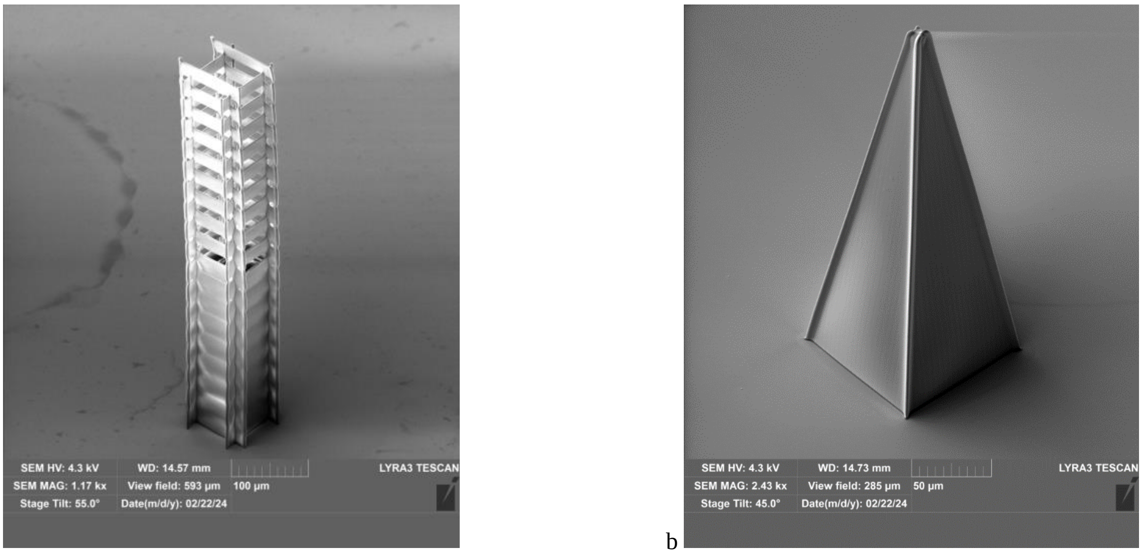


Figure 15 Grating prepared in the mr-DWL negative photoresist on glass substrate: a) overall view, b) detail cross section image of patterned resist layer

3.4. Lithographic patterning by two-photon lithography (2PL)

For two-photon lithography a femtosecond laser with a wavelength of 780 nm and objectives with 20x (NA 0.46) and 60x (NA 0.7) magnification were used at ILC and JOR. This technique was applied to demonstrate the generation of 3D patterns in up to 500 µm thick **negative tone mr-DWL resist layer**. The very small volume, called voxel, is the focal spot where the resist material absorbs simultaneously two photons during the exposure. The voxel size has here a height of 21 µm and a width of 3 µm and was chosen as the standard voxel size for the experiments, demonstrating the theoretically smallest possible resolution. The advantage of using a relatively small NA objective is that voxel size (unit of the smallest crosslinked resist polymer) is relatively large, leading to short exposure and therefore processing time, while high NA allows for higher resolution but significantly higher processing time.

Figure 16a shows a successful patterning of a 3D woodpile structure with a height of 480 µm in an originally 500 µm thick mr-DWL layer. Figure 16b shows a 200 µm tall pyramid patterns demonstrating the very smooth high-quality pattern walls. The structures exhibit well resolved details with minimal defects that could be resolved by optimizing the optical setup and laser parameters. mr-DWL resist patterns has proven to be able to have mechanically stable structures of up to 500 µm patterned in it.



a b

Figure 16. SEM image a) of 480 µm tall woodpile and b) of 200 µm tall pyramid patterns in mr-DWL

In addition, 2PL experiments have been performed on a mr-DWL layer using a 60x objective with 0.7 NA, aiming for smaller voxel size and higher resolution. The SEM images in Figure 17 show structures with a maximum height of approximately 200 µm. The top-down oriented pyramids and lenses in Figure 16a show the ability to construct undercut structures, the pyramid pattern walls in Figure 17b show smooth surfaces. The sharpness as well the accuracy of the pattern edges and the resolution of the woodpile structure needs to be improved by process parameter adjustments.

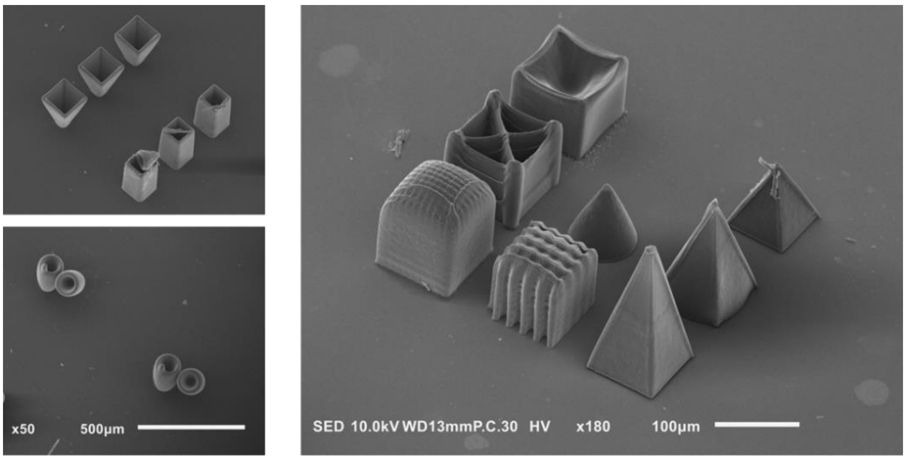


Figure 17. SEM images of 2PL patterned mr-DWL resist using 60x objective (samples tilted by 45 degrees): a) of top down oriented pyramids, towers and lenses with maximum structure height of 200 µm, and b) of a set of benchmark objects like cube, cone, woodpiles and pyramids, of which the tallest structures also reach 200 µm in height.

4. Description of the chemical, optical, mechanical, and thermal properties of the generated resist patterns

Positive resist: mr-P 22G_XP is a DNQ/novolak resist and generated resist patterns are still light sensitive and generally not suitable to be used as permanent structures. Hence, the structure formed in the positive photoresist must be transferred into a functional material for permanent application. A general challenge with very deep greyscale patterns is the photoresist still being reactive, both to light and to elevated temperatures. With the sheer amount of this reactive material in > 100 µm thick films, any pattern transfer is ought to be carried out more carefully than with thinner resists.

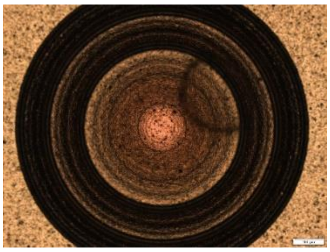
Following methods of pattern transfer were evaluated:

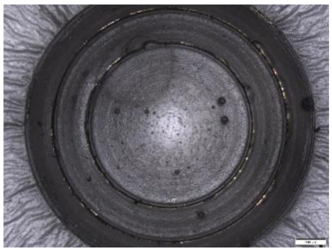
1) The resist pattern was metallised by sputtering, and then replicated by electroplating. The metal structure (negative) was used as template for UV moulding with OrmoComp® (an inorganic-organic hybrid polymer, suitable for optical application).
2) The original resist pattern was replicated in PDMS, and this intermediate pattern was transferred into the optical photopolymers OrmoComp® or OrmoClear®FX as final structures. The advantage of this process is the resist pattern remaining intact when the PDMS copy is removed, hence allowing for multiple replications into PDMS. The PDMS template itself can be replicated several times into a hybrid polymer or any other optical polymer, for permanent use.
3) The resist pattern was UV-moulded directly into OrmoComp®, thus generating a negative (opposite mold). Here, parts of the resist patterns were ripped off the substrate when the OrmoComp® replica was removed. This test was used to verify that UV moulding with an inorganic-organic hybrid polymer is possible also with deep greyscale patterns.
4) The resist pattern on a Si substrate was Deep Reactive Ion Etching (DRIE) etched using a modified BOSCH process (SPTS Rapier) having an etch selectivity of 1 to 0.84.

Greyscale pattern depths of 50 µm could successfully be replicated as described in 1). The higher patterns depth of ~ 95 µm deteriorated partly during the 9 h long electroplating process at elevated temperature. This pattern distortion was replicated in OrmoComp® in the final UV moulding step. Here, a stronger (longer) soft bake of the resist should improve the thermal pattern stability and reduce or prevent possible resist pattern distortion. Figure 18 a-h demonstrates original greyscale patterns obtained with mask aligner exposure using the HEBS glass greyscale mask and the resulting final OrmoComp® structures, without intermediate steps.

Method: **Resist pattern:** **OrmoComp® final structure:**

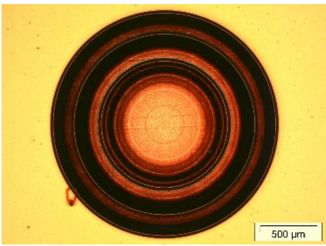
1)

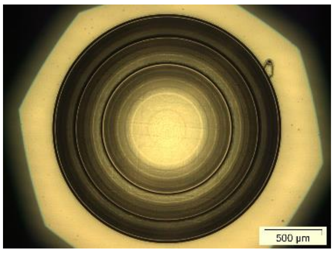
a) 

b) 

(52 µm high)

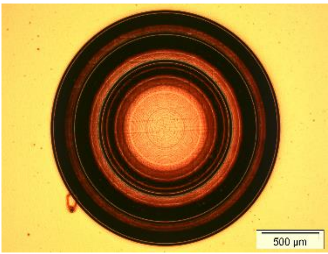
2)

c) 

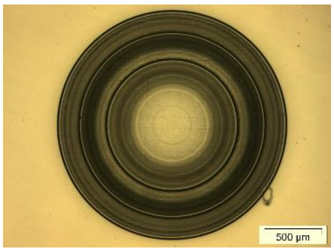
d) 

(68 µm high)

3)

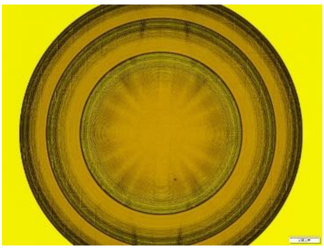
e) 

(same pattern as in 2), 68 µm high)

f) 

(negative)

4)

g) 

(16 µm high)

h) 

Etched Si substrate

Figure 18. Microscope pictures of the resist patterns after mask aligner exposure through the HEBS glass greyscale mask, and the final permanent OrmoComp® structures (b, d, f) and etched Si patterns (h) for the 4 different methods of pattern transfer

Pattern replication using method 2) was repeated with a 130 µm high greyscale pattern. Also this very deep greyscale pattern could be transferred into the final material – OrmoClear®FX – without any pattern distortion or N_2 bubble inclusion. The total thickness loss from the resist mould to the OrmoClear®FX pattern height was only 2-3 µm in different tests. SEM pictures of the original 130 µm high mr-P 22G_XP pattern, its PDMS replica, and the final hybrid polymer structure are shown in Figure 19a-c.

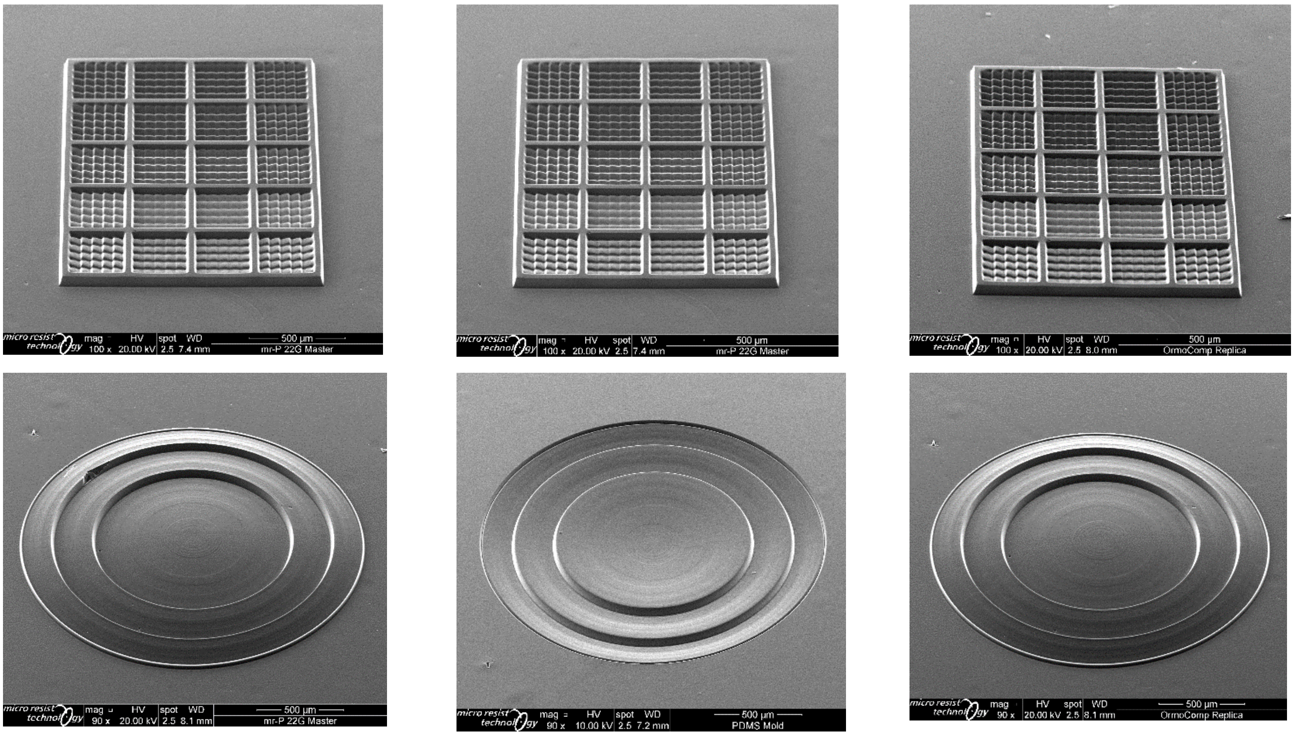


Figure 19 a. 130 µm high mr-P 22G_XP template; b. PDMS replicate and c. 128 µm high OrmoClear structure

Deep greyscale resist patterns were successfully transferred into materials suitable for permanent use – a process necessary for this type of photoresist – by four different methods. Thermal moulding of the resist patterns with PDMS followed by UV moulding with an inorganic-organic hybrid polymer suitable for optical applications was the favourite method since it was non-destructive for the photoresist template. It was successfully demonstrated that the generated positive resist master can be used to be replicated via metallisation and electroplating (pattern transfer method 1) to generate a metal shim. mr-P 22G_XP offers sufficient dry etch stability. Some defects, such as small nitrogen bubbles in the partly exposed resist patterns may lead during the vacuum based dry etching process to even larger bubbles and are visible in the dry etched Si patterns. Due to these defects, pattern transfer via a dry etching process is not the preferred patterns transfer process for thick rests film greyscale patterns.

The successful pattern transfer processes demonstrate that the thermal, mechanical, and chemical stability of the generated mr-P 22G_XP resist pattern is sufficiently stable during these pattern transfer processes.

Negative resist: The UV-vis absorption spectra of uncross linked and crosslinked mr-DWL demonstrate their absorption until of app. 425 nm, as well their transparency above that wavelength. The thermo-optic coefficient (d*n*/d*T*) describes the temperature dependence of the refractive index and is given in Figure 20 for a cured mr-DWL layer. Thermo-optic coefficient is calculated as $dn / dT = (-6.8 \times 10^{-5} \pm 1.1 \times 10^{-5})\ K^{-1}$.

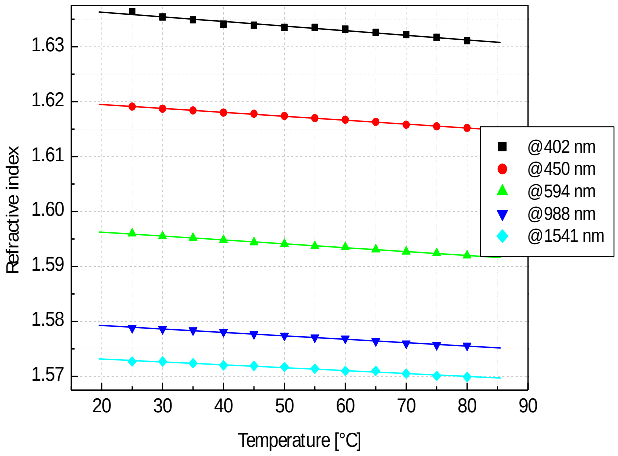


*Figure 20.*Dependence of the refractive index of cured mr-DWL on the temperature; film thickness 2 µm, standard processing

The highly crosslinked mr-DWL resist patterns are known to offers high chemical, thermal and mechanical properties and mr-DWL patterns are favoured to be used as permanent material.

5. Conclusions

Positive resist layers up to (190 – 200) µm can be generated by the use of the newly developed mr-P 22G_XP 2nd Gen prototype resist formulation as targeted within OPTIMAL project. Lithographical patterning parameters are developed for the up to 150 µm thick resist layers. With the current mr-P 22G_XP resist formulation high precision 2.5D greyscale structures, exposure depths of up to 150 µm by one-photon lithography (1PL) and an achievable surface roughness on patterned resist surface of about 33 nm were reproducibly and defect-free obtained. High quality high aspect ratio (AR) mr-P 22G_XP gratings on substrates of an area of 25 x 25 mm^2 by laser interference lithography (LIL) demonstrates the high grating homogeneity and quality.

Deep greyscale resist patterns were successfully transferred into materials suitable for permanent use – a process necessary for the positive photoresist. Thermal moulding of the resist patterns with PDMS followed by UV moulding with an inorganic-organic hybrid polymer suitable for optical applications was the favourite method since it was non-destructive for the photoresist template. The successful pattern transfer processes demonstrate that the thermal, mechanical, and chemical stability of the generated mr-P 22G_XP resist pattern is sufficiently stable during these pattern transfer processes.

With mr-DWL negative resist series up to 180 µm thick resist layers can be generated in a single coating step. Up to about 500 µm thick layers can be generated by the use of mr-DWL_100 or the newly developed mr-DWL_120_XP with even higher viscosity (as 2nd Gen material) by a specific weight-controlled coating step. Lithographical patterning parameters are developed for the up to 500 µm thick resist layers. Up to 500 µm thick resist patterns were successfully generated by the use of mask based exposure using mask aligner (broadband) or UV-LED @ 405/410 nm. With the current mr-DWL resist formulation resist patterns with a maximal height of up to 130 µm are obtained when laser direct writing as one-photon lithography (1PL) with a single emission @ 405 nm is applied. The successful patterning of a 480 µm thick 3D pattern by the application of two photon lithography (2PL) with a voxel size of about 21 µm in height and 3 µm in width in a 500 µm thick mr-DWL layer was successfully demonstrated.

The highly crosslinked mr-DWL resist patterns are known to offers high chemical, thermal and mechanical properties and mr-DWL patterns are favoured to be used as permanent material.

6. Degree of progress

This document describes (Chapter 3) the properties of lithographic patterning for a positive & a negative tone resist series for the generation of binary (2D), greyscale 2.5D and 3D patterns by using 1) a mask based aligner process, 2) one photon laser (1PL) & a two/multi photon laser lithography process (2PL) and 3) laser interference lithography process (LIL). In addition (chapter 4) the summary of the chemical, optical, mechanical, and thermal properties of the generated resist patterns is reported. Thus, the degree of progress of the Deliverable D1.2 is 100%.

7. Dissemination level

The contents of this Deliverable are publishable and, after the approval from the European Commission, will be made available to broad public, published in the OPTIMAL project website (page “results”) and in the Cordis’s platform.