APPLICATION NOTE

FABRICATION OF REFRACTIVE MICRO-LENSES FOR ORBITAL ANGULAR MOMENTUM GENERATION BY DIRECT LASER WRITING AND NANOIMPRINT LITHOGRAPHY

by

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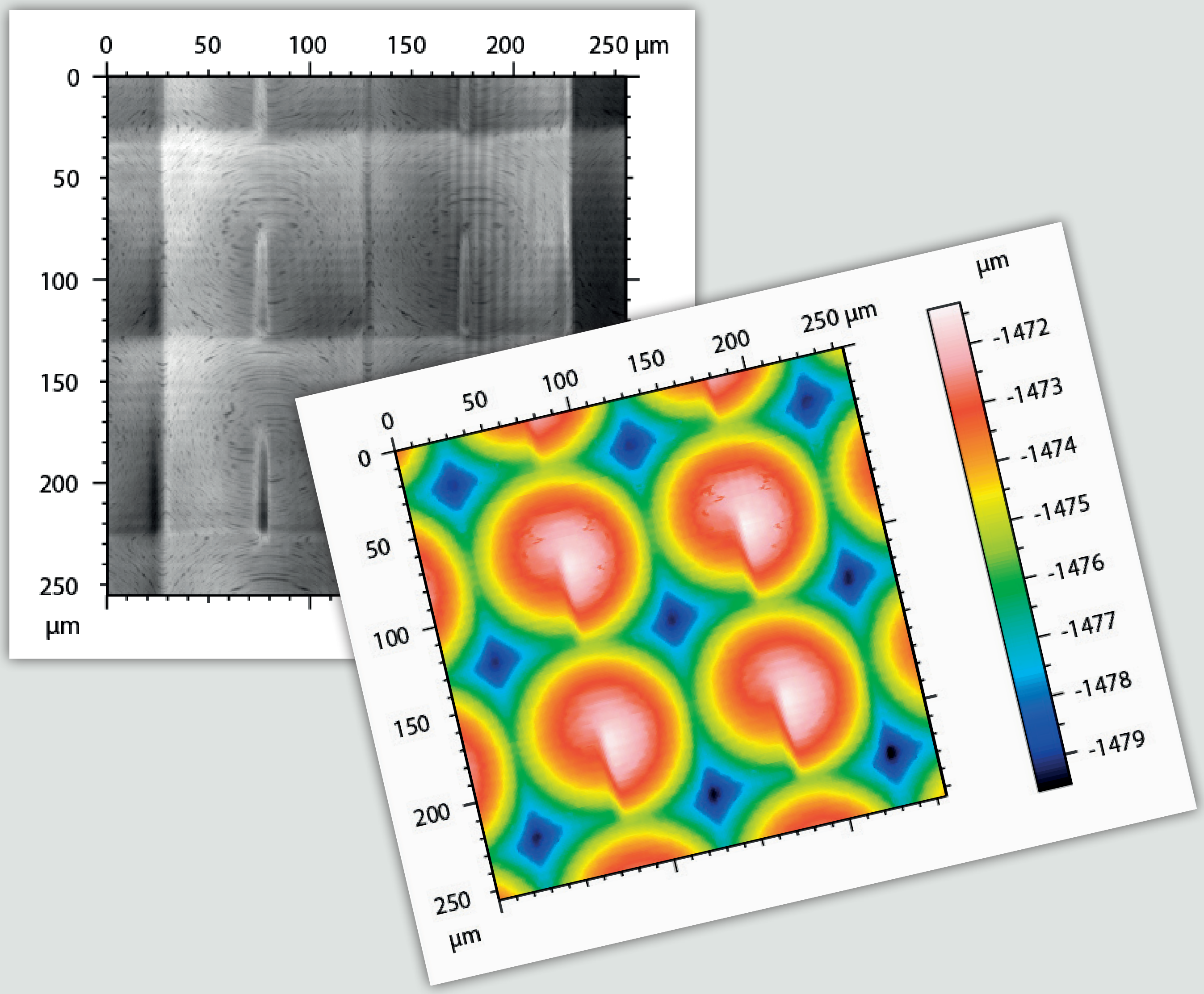

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1. INTRODUCTION

Although Diffractive Optical Elements (DOE) and metasurfaces are central in today's photonics research, "old" refractive optics are still key elements in many practical applications. In fact, while the prospects of new ultracompact devices with unprecedented characteristics is fueling the interest towards DOE and metasurfaces, their practical application often lags behind due to technical limitations. On one side DOE suffer from low diffraction efficiency, strong chromatic aberration and undesired "ghost" images from unsuppressed high diffraction orders, on the other side metasurfaces are still in the process of technological maturation and challenging in terms of design and fabrication.

While DOEs and metasurfaces are implemented as binary or multilevel nano-structures, refractive micro-optics are smooth grayscale micro-structures, often referred to as "2.5D".

In the fabrication of refractive micro-optics with smooth profile, laser writing and nanoimprint lithography appear as a winning combination, the first enabling the origination of grayscale patterned surfaces up to wafer scale, the second capable of small, medium or large volume productions from a single origination by accurate replication of patterns into suitable functional materials.

In general, the shape of a micro-optical element originated by direct laser lithography can be optimized within a few iterations, to reach the target precision, usually a fraction of the wavelength at which the optics is supposed to work.

In this Application Note we will discuss the fabrication of array of refractive elements to generate electromagnetic waves carrying Optical Angular Momentum (OAM). This optical function has been previously implemented in various ways including pairs of accurately aligned cylindrical lenses, Spiral Phase Plates (SPP), static or dynamic DOE (where the dynamic version is obtained by liquid-crystal spatial light modulators), or more recently by metasurfaces. However, other elements are usually inserted downstream in the optical path to counteract the natural divergence of light beams carrying OAM modes or to exploit their properties in application requiring focusing, such as for in-coupling the OAM in optical fibers for or waveguides in telecommunication or for setting biological cells or bids floating in liquid into rotation by optical tweezers.

In this Application Note we describe shortly the fabrication process of an optical element that can be understood as the superposition of a Spiral Phase Plate and a spherical micro-lens, so as to integrate in a single element the functions of transferring an orbital angular momentum to an incoming plan wave and focusing it to a small ring at a given focal distance.

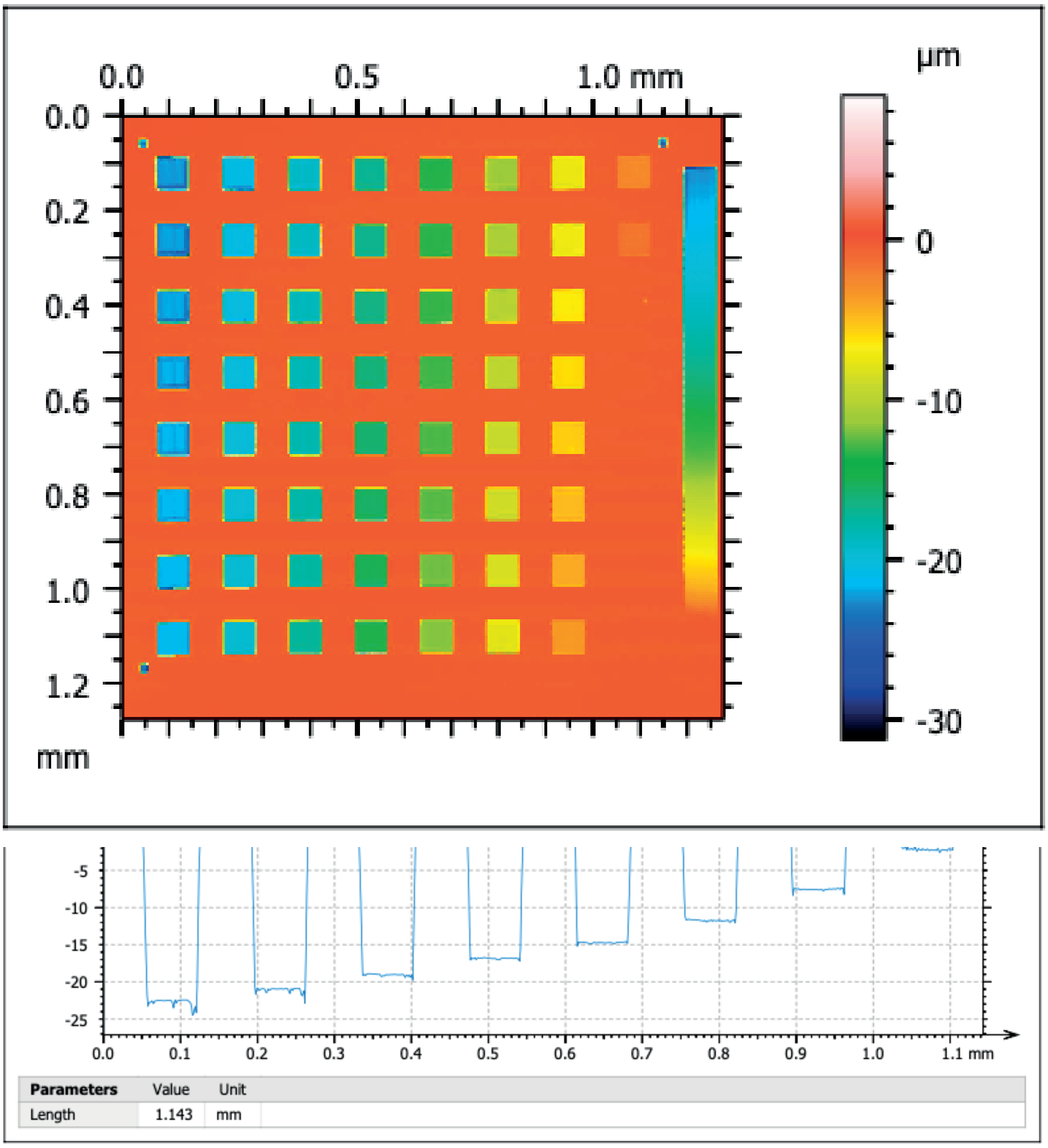
However, the general scheme used in this fabrication process is not limited to the present case, but can be taken as a model in a variety of other refractive micro-optics.

The reported work has been obtained using a Heidelberg Instruments DWL 66$^+$ laser writing system and a Germanlitho GL PS01 UV-NIL equipment.

2. CONTRAST CURVE MEASUREMENT

It is known that for given conditions of film preparation and development, the dissolution rate of a positive photoresist depends on the local density of photons of suited energy absorbed in the volume during the exposure. This volumetric density of absorbed photons is proportional, as a first approximation, to the areal density of photon impinging on the surface. This approximation overlooks the fact that direct laser writing involves several proximity effects, leading to a broader (non-local) background illumination (e.g. the backscattered photons from the focused laser beam at the resist substrate interface in case of a highly reflective substrate).

The behavior upon exposure of a photoresist can be captured by a contrast curve, which measures the profile depth reached after the development as a function of the areal photon density (or as a function of a parameter directly correlated to it, such as gray-value distribution (GVD), linear by definition, that is fed into the laser writing machine to control the amount of radiation delivered according to the local gray-values map). The contrast curve can be measured in practice by exposing at different dose levels an array of elements, for example an array of squares, from which it is possible to extract the relation between the gray-values of the map and the depths measured by tools such as an AFM, a mechanical profilometer or a confocal microscope.



Parameters	Value	Unit
Length	1.143	mm

Figure 1, (top) Confocal map of the “dose matrix” exposed on a positive photoresist to measure the contrast curve; (bottom) cross-section profile taken across the top row of the squares.

Once this correspondence is measured, the geometrical shape of a newly designed optical element can be translated into the areal photon density that need to be delivered to the photoresist to produce that given shape and can be represented by a grayscale map controlling the laser writer in the writing process.

The Heidelberg Instruments DWL 66$^+$ laser writer allows to discretize the exposure in up to 1024 grayscale levels (gray-values). In order to determine the contrast curve we sample here 64 different experimental points (each experimental point being the average of hundreds of data referring to the same exposed square) and interpolated with a polynomial function, as show in fig 1.

The accurate measurement of the contrast curve is the key step that enables designing and realizing with excellent approximation even complex optics already from the first attempts.

3. PHOTORESIST FILM AGING

In our experience there is a second key ingredient in a successful laser writing process. A special care is required in photoresist film preparation and conditioning, as reproducibility is strongly affected by preparation and by aging of the photoresist film (not to be confused with the aging linked to photoresist shelf life).

In fact, after spin-coating and annealing the film is strongly out of equilibrium with the environment and needs some time, hours to days, to reach a steady behavior in response to a writing process. The main phenomenon occurring during this time is the film rehydration that strongly depends on the cleanroom environment and stability. Only after such time, dependent on resist formulation, film thickness, and clean-room environment, exposure results become more controlled and reproducible. Since process optimization can occur over several days, it is essential to keep in mind that initial results are affected by a drifting behavior of the photoresist.

We checked this fact in detail for AZ4562 and ma-P1275 photoresists by dicing spin-coated wafers and using for the tests pieces from the same preparation at different times, which lead us to observe a significant drift in the contrast curve, as shown in figure 2.

AZ4562, 26 MM THICK FILM.

We estimate the rehydration time by exposing a “dose matrix” pattern at different time intervals after annealing. AZ4562 photoresist was spin coated in a single deposition step at 450 rpm for 60 seconds and baked for 15 minutes at 100°C, resulting in 26 µm film according to ellipsometry measurements. The wafer was kept in the room with stable humidity of 55% and temperature of 19°C for two hours and then we expose the first sample. The next exposures were done 4, 9 and 28 hours after baking. To exclude any possibility of uncontrolled influence by other parameters we used, for all the exposures, small pieces diced from the same wafer.

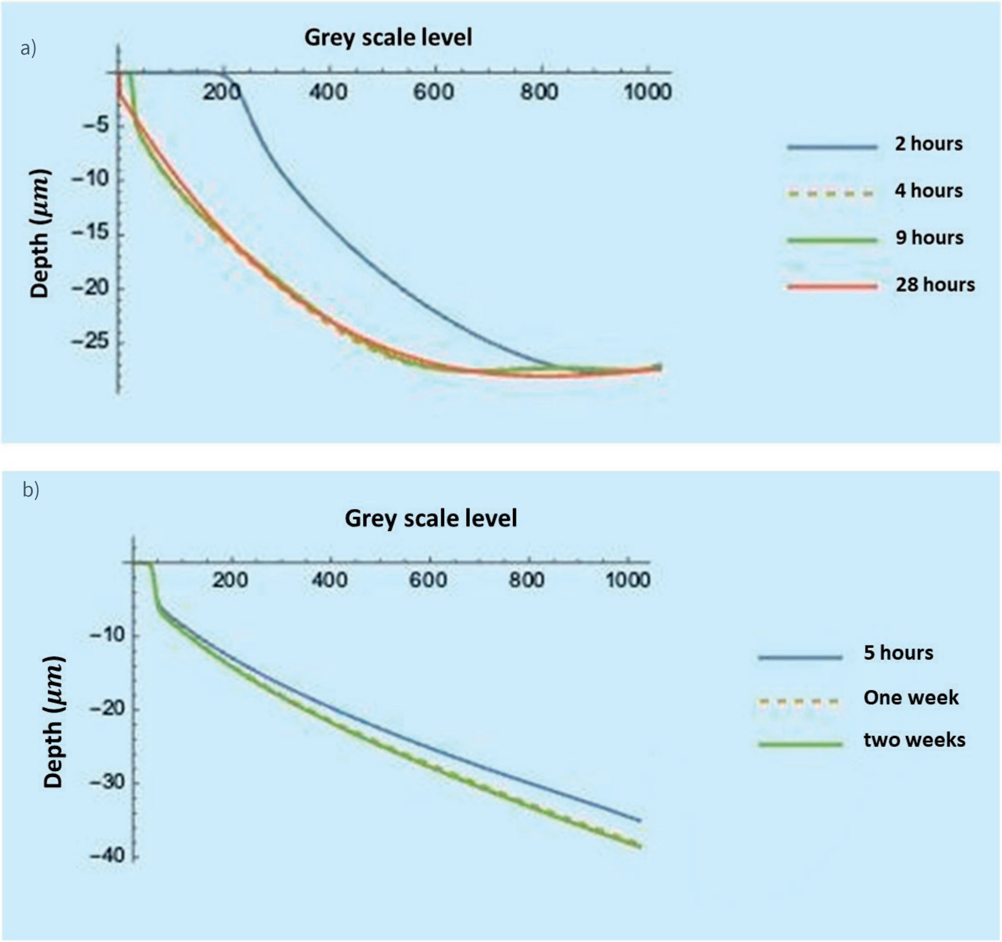


Figure 2, a) the contrast curves relative to a 26 µm thick film of AZ4562 exposed at different times from soft bake of the film. b) the contrast curves for ma-P1275 exposed at different times from soft bake.

The laser exposure parameters were identical in all exposure parameters (focus:0, Intensity: 100%, laser power: 90 mW and filter: 5%. A solution of AZ 400K: water 1:4 by volume was used to developed the exposed structure for 15 minutes under manual shaking of the container – automated agitation being advisable for increased repeatability. By analyzing the contrast curve, the time for rehydration is considered as the delay from film annealing after which no considerable variation of the curve occurs. The design of the lens is converted with n-over 20. We use a wafer with the same preparation condition as we did for dose matrix. The delay time from baking the wafer until the exposure is more than 24 hours and the lens is exposed with focus: 0, Intensity: 100%, laser power: 60 mW and filter: 5%. The AZ 400K diluted 1:4 by volume with water and the exposed structure developed 8 minutes with occasional rotational movements.

MA-P1275, 40 MM THICK FILM.

We investigated the rehydration time of ma-P1275 photoresist using dose matrix structure and exposing the structure 5 hours, 1 week and two weeks after preparation. All the samples are used from the same wafer and stored in room condition with humidity of 42% and temperature of 20°C. The wafer is spin coated with spinning velocity of 450 rpm for 60 seconds and baked at 60°C for 15 minutes and at 100 °C for 10 minutes. The AZ 400K diluted 1:4 by volume with water and the exposed dose matrix structure is developed 10 minutes with occasional rotational movements.

Figure 2a illustrates the contrast curves for AZ4562 at different times steps after baking where the contrast curves shown in panel b belongs to ma-P1275 resist.

4. DEFINITION OF THE OPTICAL ELEMENTS

An optical element can be defined mathematically as a (single-valued) function z=h(x,y). In general, all surfaces that can produced in a direct laser writing process do not have overhangs, and can thus be described in this way. Also, the class of structures that can also be replicated by Nanoimprint Lithography cannot have overhangs, since this would result in the disruption of the structures during de-molding. As a consequence, what can be originated by Direct Laser Writing can be replicated by Nanoimprint Lithography. The function z=h(x,y) can be directly derived from the phase map that the optical element has to impart to imping light to perform the desired optical function. In the present case, we wanted to obtain elements generating an array of optical vortices and at the same time concentrating the e.m. field at given focal distance. This can be obtained by optical elements that are the superposition of a helix with a spherical lens, whose mathematical expression can be represented by the following equation,

$$\text{hradial}(r,\phi) = \frac{\sqrt{(n^2-1)\left(f^2-\left(\frac{\lambda m n \phi}{2\pi}\right)^2+r^2\right)+\left(f-\frac{\lambda m n \phi}{2\pi}\right)^2}}{1-n^2}$$

where f is the focal distance, n is the refraction index of the material of the lens, lambda is the wavelength, r and φ are the radius and the azimuthal angle of a cylindrical reference system, m is the optical paths delay in units of wavelengths accumulated at the step discontinuity.

The mathematical description of the element is then mapped onto a regular grid, corresponding to the pixel size chosen during the exposure and the height of the profile of the structure (translated in order to represent the height below the surface of the resist) is converted into a discretized map of grayscale value using the contrast curve measured previously. The grayscale values in the map are proportional (with a proportionality constant factor that depends on the settings of the laser writer) to the local number of photons per unit area delivered pixel by pixel in which the surface is divided.

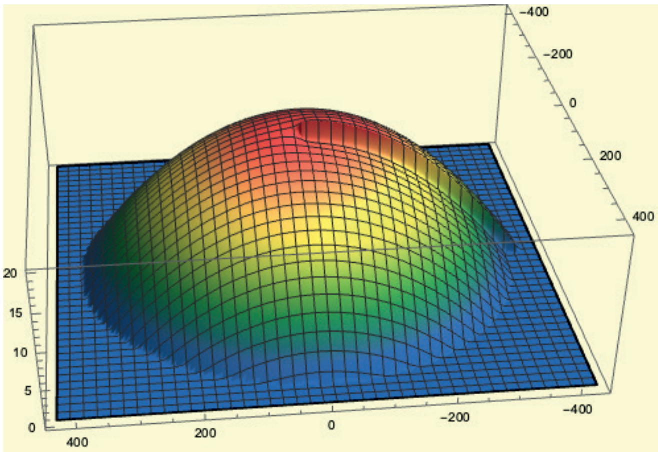


Figure 3, Example of microlens for OAM generation and focusing, for 500 nm wavelength, n=1.5, m=-6, f=250 µm.

5. FROM THE PROTOTYPE TO THE UV-NIL REPLICAS

The Direct Laser Writing process was carried on AZ4562 photoresist the 26 µm film prepared as described in section 2. Contrast curve measurement. The film was used after one day from the spin-coating and annealing. After exposure (focus:0, Intensity: 100%, laser power: 90 mW and filter: 5), the photoresist was developed in a solution of AZ 400K : water 1:4 by volume for 8 minutes, according to the condition used for measuring the contrast curve. The master wafer shown in figure 4a, and the imaging results is demonstrated as a 2D map on the upper left and 3D model on the upper right in figure 5a. The structure is characterized by taking topographic maps with a Zeiss AXIO, imager Z2 Vario confocal microscope with a 50X objective lens.

The next step is the replication of the structures, which can be carried out by nanoimprint lithography and is done for two main reasons. The first is to produce in a short time many copies of a micro or nanopattern that would be time consuming to make by direct writing process. The second is to decouple the material properties of the final material from those of the photoresist used to form the shape by laser writing.

The replication of the originated structures is carried out in two steps, from the master to a working stamp and from the latter into a final optical device. For the replication of the structures from a master we proceeded as follows. The structure weas first replicated (with the negative profile) into a UV-curable resin to be used as a "working stamp" (figure 4b). The process was performed in a Germanlitho GL MRT UV nanoimprint equipment tool. The master originated by direct laser writing process was fixed on the stage of the tool and a small amount of PS01 photoresist was poured on it. A PET sheet approaches slowly to the master surface until the contact with the resin is established. Then, the distance is progressively reduced until the resin spreads onto the full surface of the wafer filling in the structures. When this process is complete, the photoresist is exposed to 365 nm UV light, with 54 J/cm^2 total irradiation. After slowly separating the PET sheet from the master the negative pattern of the structure (figure 5b) is protruding in the cured GL PS01 on the PET sheet as a carrier.

To replicate the structures from the working stamp into an array of optical devices we proceeded analogously, with the same tool, but using for the final step a different Germanlitho resin, the HS photoresist. The process was carried out with exposure of 36 J/cm^2 on a 4inch soda-lime glass substrate with 1 mm thickness. The resulting structure is shown at figure 5c.

6. CASE OF BINARY STRUCTURES

The above scheme can be applied also to binary structures, such as in the example shown below. Instead of using a resist with a low contrast (like AZ4562, suited for grayscale lithography), for binary structures it is more convenient to use resist with high contrast. To produce an 800 nm period grating, we choose to use SPR-220, that has a contrast curve as shown in figure 6.

We spin coated SPR220 diluted 1:1.5 with PGMEA. The photoresist is spin coated at 4500 rpm spinning speed on a soda-lime glass substrate, resulting in a 350 nm thick film, followed by soft-bake at 115 °C for 90 s.

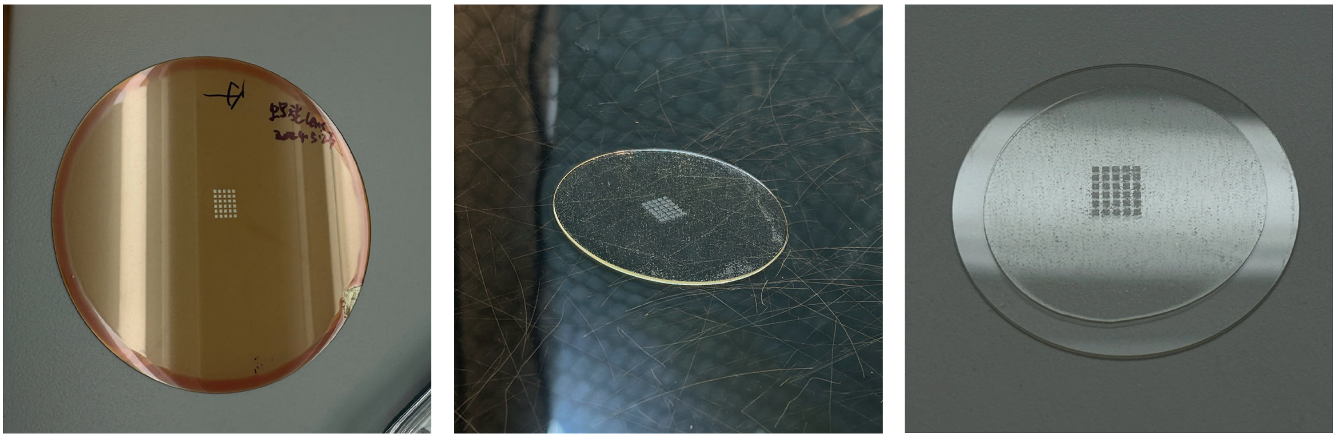
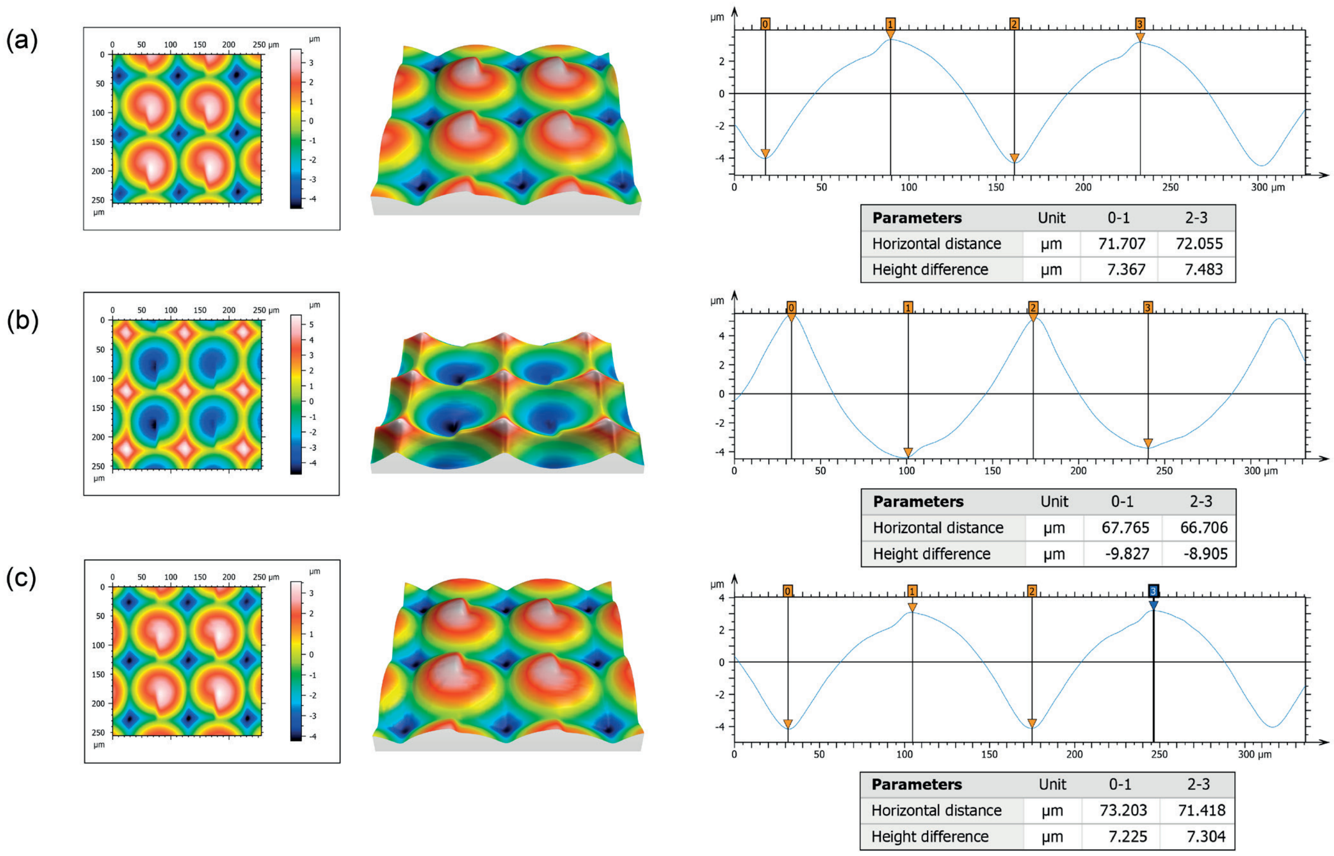

a) b) c)

Figure 4, a) the master originated by direct laser writing. b) the negative of the structure on PET sheet (working stamp). c) the final replication of the structure.



(a)

Parameters	Unit	0-1	2-3
Horizontal distance	µm	71.707	72.055
Height difference	µm	7.367	7.483

(b)

Parameters	Unit	0-1	2-3
Horizontal distance	µm	67.765	66.706
Height difference	µm	-9.827	-8.905

(c)

Parameters	Unit	0-1	2-3
Horizontal distance	µm	73.203	71.418
Height difference	µm	7.225	7.304

Fig. 5 The image shows the 2D (left) and 3D reconstruction (right) of produced micro-optical elements. a) by direct laser writing process, b) replicated into a “working stamp”, c) reproduced as a final device in a different optical material by UB-NIL process.

The design was exposed using the HiRes Write Mode of the DWL 66^{+}, a lens with a large numerical aperture and a focal distance of 2 mm, and the following exposure parameters: Intensity: 100%, Filter: 1%, laser power 115 mW. The exposed film underwent a post-exposure annealing at 115°C for two minutes and the structures were revealed by a development of 30 s in diluted TMAH 1:3 with water. Figure 7a, shows well resolved grating in the original laser written pattern (Master).

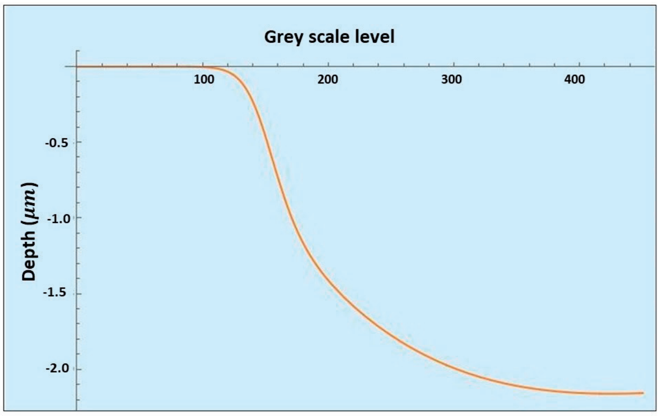
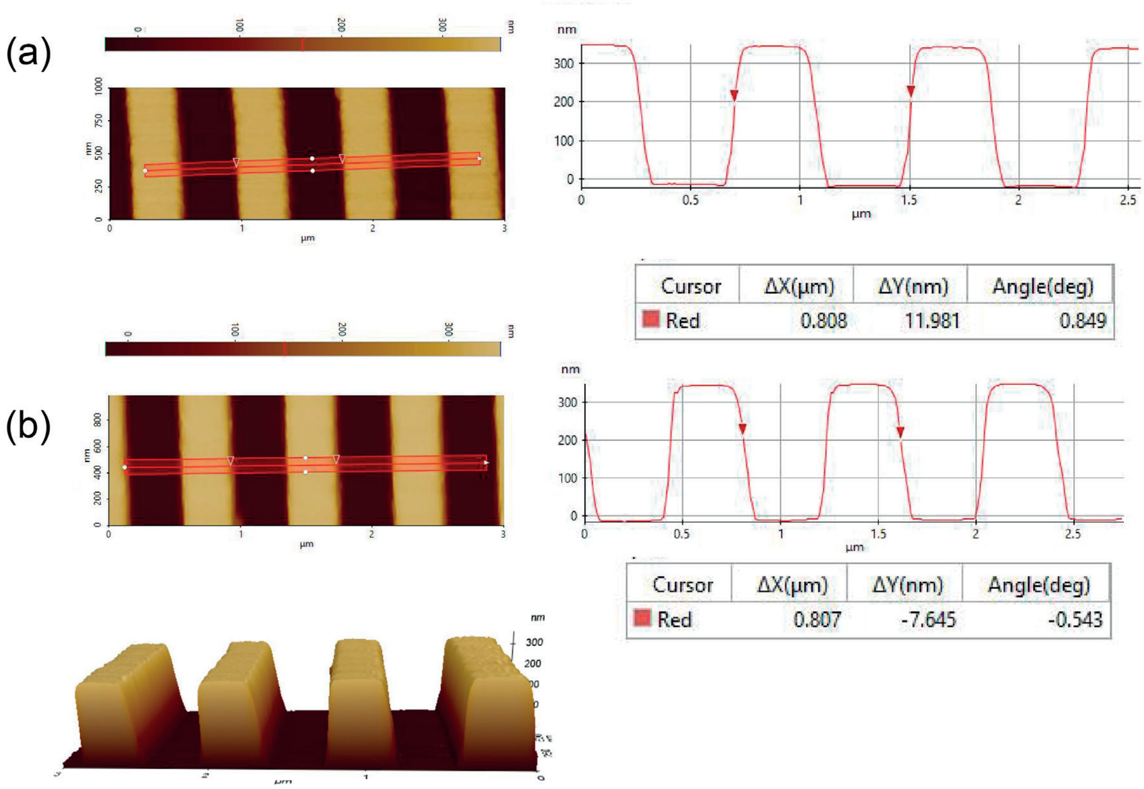


Figure 6, the contrast curve of SPR-220 film.

For replication of the structure, we choose the roll to plate process using a 12-inch CLIV Germanlitho nano imprinting tool (figure 8). The working stamp is prepared with the same technique as explained at section 6. To replicate the structure from the working stamp, the photoresist (R V series, Germanlitho) is spin coated onto the desired substrate followed by baking. The detailed description of this process is outside the scope of this Application Note. Essentially, the PET sheet stretched on a frame with a controlled pulling force is placed at a small distance from the substrate. A cylinder is set to roll onto the PET sheet at a controlled speed, to produce a uniform contact of the stamp to the substrate. When the full conformal contact is obtained, the photoresist is cured by UV exposure (365 nm, I-line, 36.0 J/cm^{2}) and then the working stamp is separated from the replicated structure on the substrate.



Cursor	ΔX(µm)	ΔY(nm)	Angle(deg)
🟥 Red	0.808	11.981	0.849

Cursor	ΔX(µm)	ΔY(nm)	Angle(deg)
🟥 Red	0.807	-7.645	-0.543

Figure 7, a) Fabrication of binary grating of 800 nm period by laser writing process and b) the Nanoimprinted final replication. The intermediate “working stamp” is omitted.

7. CONCLUSIONS

We have demonstrated on a specific optical element, the array of microlenses to produce focused light waves carrying Orbital Angular Momentum. We showed the detailed sequence of process steps that allows to produce a broad variety of micro-optical elements based on Direct Laser Writing and UV-NIL Nanoimprint Lithography. The correct shape is of outmost importance for the precise implementation of optical functionalities of refractive optics. For this purpose, the preliminary work for accurate measurement of the contrast curve, the aging behavior of the resist film to ensure the reproducibility of the results in the laser writing process, is an essential step in the development of the process. Laser writing process can also be used to produce high resolution structures by using resist with higher contrast compared to the case of grayscale 2.5D structures. We finally showed how NIL can be used to replicate into other functional materials the pattern through the use of “intermediate working stamp” to produce the final functional devices.

Figure 8, Germanlitho 12-inch CLIV nano imprinting tool.

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