

Research paper

# High-resolution grayscale patterning using extreme ultraviolet interference lithography



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## ABSTRACT

Grayscale patterning is technologically relevant in the fabrication of micro-optics elements, microfluidics, micro-electromechanical devices, to name a few. So far, the state-of-the-art is limited to micrometric scale, which is of interest for optical applications in the visible spectrum. In this work, we used extreme ultraviolet light and an interference lithography method to demonstrate the feasibility of grayscale patterning with high lateral and vertical resolution. A double exposure was carried out on poly(methyl methacrylate) photoresist using periodic lines/spaces of two different pitches (100 nm and 50 nm). The resulting morphology of photoresist after development, analyzed by scanning electron microscopy and atomic force microscopy, showed that a dense one-sided blaze profile consisting of three grayscale levels was obtained. The equivalent groove density of the blazed grating was 10,000 lines/mm, which is a remarkable achievement of significant interest for applications such as high resolution and high efficiency diffraction optics. Furthermore, combinations of overlay from non-multiple pitches (100 nm and 80 nm) was accomplished and unconventional structures with nested trenches with total period of 400 nm were obtained.

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## 1. Introduction

There is a growing number of applications where grayscale patterns are needed, such as micro-optical interconnects [1], kinoform lenses for X-ray focusing [2], microfluidics channels of variable geometry [3], micro-electromechanical devices [4], optical diffusers [5], and biomimetic structures [6,7]. The ability to fabricate such microstructures by the so-called grayscale lithography has been an enabling technology for the realization of on-chip optical-to-electronic converter, blazed optical lenses with higher efficiency than conventional binary gratings, functional micro- and nanofluidics channels, and, in general, the manufacturing of components to a larger scale of integration. In conventional lithography, the designed exposure dose is constant and it is sufficiently high to either completely remove the photoresist from the exposed parts (in a positive-tone resist) or to leave it unaltered in the unexposed areas. The resist image remaining after development is, therefore, binary. On the contrary, in grayscale lithography the exposure dose is changed across the sample and within the pattern: as a result, the remaining resist shows a thickness gradient and has the morphology of a 3-dimensional surface. The lithographic contrast of the resist is an important parameter in grayscale lithography, because it has to be

adjusted in order to finely tune the remaining resist thickness as a function of exposure dose.

A variety of lithographic techniques are nowadays state-of-the-art to realize grayscale lithography. In variable-dose direct writing, the dose is tuned during the raster exposure of a beam (typically, an electron beam or ion beam), which is relatively straightforward to attain by using modern beam controllers. The use of an electron or ion beam brings the same advantages and disadvantages as in conventional lithography, specifically the versatility of a maskless system, the high resolution, and relatively low throughput. Another possibility is the use of optical projection systems where the constant exposure dose is filtered by a mask featuring varying transmissivity levels. This technique has been implemented using optical sources in the visible range [1], in the ultraviolet [8], and in the X-rays [9] and goes under the name of grey-tone lithography. Finally, scanning probes can also be used to produce multi-level resist patterns, provided that the exposure dose can be controlled by spatially tuning the thermal [10] or thermochemical [11] gradient across a suitable sensitive material. Recent technological advances make scanning probe the highest resolution patterning method (about nanometric vertical resolution and about 30 nm lateral resolution). Nevertheless, scanning probes are unable to fabricate large areas in a reasonable time and can only make use of a limited choice of resists.

The abovementioned studies demonstrate that the resolution of a resist patterned in grayscale mode strongly depends on the lithographic

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tool. Feature size is determined by the maximum patterning detail attainable across the thickness gradient (vertical dimension) and along the surface (lateral dimension). The former depends on the resolution in the dose control, which is typically very high in a digital high-frequency beam controller, in an optical shutter or in a Kelvin mode scanning probe. The latter is related to the lateral resolution of each tool which is determined by either the diffraction limit of light, the electron beam spot size and proximity effect or the radius of the probe tip. In this regard, an additional limitation to the lateral resolution is determined by the fact that the grayscale process is tuned for low lithographic contrast. As a result, when creating a grayscale pattern by electron beam lithography, the proximity effect (due to electrons backscattered from the substrate) has a considerably more detrimental effect than it has in conventional lithography. Furthermore, at increasingly high resolution, exposed and unexposed areas are more closely packed together and the resulting resist profile after exposure is not as sharply defined as it was in the design (which cannot be compensated for by the software). In sum, dense grayscale patterns fabricated by electron beam lithography attained a remarkable vertical resolution of  $\sim 1$  nm, but only about 100-nm-wide features along the lateral dimension [2,5], despite the availability of proximity effect corrections also in 3D geometry [12]. The smallest feature size achieved for blazed gratings fabricated by the variable direct writing of a Ga ion beam has been down to 200 nm pitch [13]. Structures realized by optical exposure tools or digital micromirrors are strongly limited by the wavelength used, typically deep-UV at 193 nm. So far, high lateral resolution grayscale patterns have been achieved only by scanning thermal probes, with the smallest features size of about 30 nm [14]. In that case, the lateral limitation was dictated by the tip radius; the vertical range was instead limited by the maximum effective depth of the heating.

In this work, the aforementioned limitations are circumvented by the use of extreme ultraviolet (EUV) optical lithography. EUV light, at 13.5 nm wavelength, enables significantly higher resolution than conventional optical lithography at 193 nm wavelength. In fact, EUV lithography is the leading candidate for high-volume manufacturing for the next generation very large scale integrated (VLSI) circuits. The use of photons also brings the advantage of no substrate charging and no proximity effect during exposure.

We present here a new methodology based on multiple overlay exposures by means of an EUV diffraction mask, featuring periodic patterns of different periodicity to obtain grayscale lithography. Using this approach, we effectively bypass the main limitations to achieve high-resolution grayscale lithography and thereby produced 100-nm-pitch blazed gratings with 3 grayscale levels in an echellette geometry. Furthermore, our strategy makes use of existing infrastructure and technology of extreme ultraviolet interference lithography (EUV-IL) and does not add to the complexity of the exposure tool. The gratings obtained in this work have a density of 10,000 lines/mm and have potential applications as, for example, high spectral resolution diffraction reflection gratings or diffusers in the visible range and shorter wavelengths.

## 2. Experimental details

Polarized EUV light at 13.5 nm wavelength with a spectral resolution of 4% and a flux of about  $30 \text{ mJ/cm}^2$  was generated from the Swiss Light Source synchrotron. The exposure tool was installed in the XIL-II beamline and consisted of a vacuum chamber equipped with a motorized stage (3-axis movement). The exposure tool could be fitted either with large frame aperture ( $0.5 \times 0.5 \text{ mm}^2$ ) for flood exposures or with interference masks to perform EUV-IL of periodic lines/spaces patterns. In the first case, flood exposures were employed to find the suitable process conditions. To this purpose, the resist was exposed to increasing dose of EUV light and, after development, the remaining thickness was measured by a Dektak stylus profilometer (Bruker). The remaining thickness,  $t$ , was then normalized to the initial thickness of the resist

and plotted as a function of the dose,  $D$ , to obtain the so-called resist contrast curve. Afterwards, a dose response function of the type:

$$t(D) = \frac{1}{1 + e^{\gamma(D_0 - D)}} \quad (1)$$

was fitted to the experimental data and the value of the two parameters ( $D_0$ : the dose-to-clear, and  $\gamma$ : the lithographic contrast) was hence extracted. Mathematically, the former parameter represents the dose at the inflection point and the latter represents the natural-log slope of the curve at dose  $D = D_0$ .

Poly(methyl methacrylate), PMMA, is a positive tone photoresist, sensitive to a broad range of wavelengths. PMMA is also easily treatable by thermal reflow, which is a convenient treatment to optionally smooth out its surface after lithographic patterning [15]. A commercially-available PMMA solution of molecular weight 120 k (as provided from manufacturer *micro resist technology* GmbH), normally yields about  $1 \mu\text{m}$  thick film, when spin coated at 3000 rpm speed. This commercial solution was further diluted (using anisole, the same solvent as in the original casting) to obtain a coating of 80 nm thickness at 3000 rpm for 60 s. A post application bake was carried out at  $140^\circ\text{C}$  for 120 s to evaporate the casting solvent from the film. Four development conditions were explored, using different combinations of methyl isobutyl ketone (MIBK), isopropyl alcohol (IPA) and deionized water (DIW). In all these conditions, the development time was 30 s, followed by rinse for 30 s in either DIW or IPA. Development and rinse were always conducted with solutions at room temperature.

The contrast curves and the corresponding dose response fit for all the four development conditions are shown in Fig. 1. The dose-to-clear  $D_0$  and the lithographic contrast  $\gamma$ , extracted from the fit, are summarized in Table 1. It is clearly noticeable that the shape and slope of the plot depend strongly on the choice of developer/rinse combination. Previous works have explored the effect of various developers on PMMA in more detail and our findings are in excellent agreement with those [16].

In the scope of this work (i.e., the minimization of the lithographic contrast for grayscale patterning) the lowest value of  $\gamma = 0.44$  was achieved by choosing the development with the MIBK:IPA 1:1 mixture, followed by IPA rinse. This process condition was therefore used for the processing of all samples discussed in the following sections. It was also found that the use of DIW in the developer mixture drastically reduced

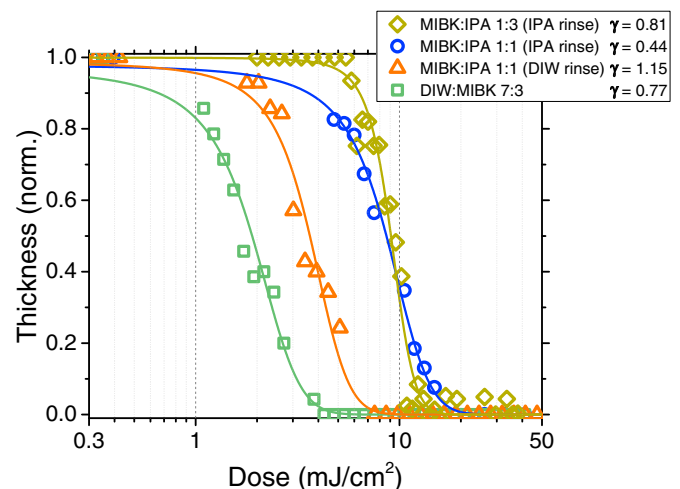


Fig. 1. Normalized contrast curves of PMMA at EUV, processed with four different developer/rinse combinations: MIBK:IPA 1:3 with IPA rinse (yellow diamonds), MIBK:IPA 1:1 with IPA rinse (blue circles), MIBK:IPA 1:1 with DIW rinse (orange triangles) and DIW:MIBK 7:3 with IPA rinse (green squares). The corresponding best fitting dose-response curve are also shown (lines). The extracted lithographic contrast,  $\gamma$ , is reported in the legend.

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