Phase holograms in polymethyl methacrylate

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(Received 27 May 1992; accepted 6 August 1992)

Complex computer generated phase holograms (CGPHs) have been fabricated in polymethyl methacrylate (PMMA) by partial exposure and subsequent partial development. The CGPH was encoded as a sequence of phase delay pixels and written by e-beam (JEOL JBX-5DII), a different dose being assigned to each value of phase delay. Following carefully controlled, partial development, the pattern appears, rendered in relief, in the PMMA which then acts as the phase-delay medium. The exposure dose was in the range 20–200 μC/cm^2, and very aggressive development in pure acetone led to low contrast. This enabled etch depth control to better than ± 20 nm corresponding to an optical phase shift in transmission, relative to air, of $\pm\lambda_{\mathrm{vis}}/60$. That result was obtained by exposing isolated 50 μm square patches and measuring resist removal over the central area where the proximity effect dose was uniform and related only to the local exposure. For complex CGPHs with pixel size of the order of the proximity radius, the patterns must be corrected for proximity effects. In addition, the isotropic nature of the development process will produce sidewall etching effects. The devices fabricated were designed with 16 equal phase steps per retardation cycle, were up to 3 mm square, and consisted of up to 10 million 0.3–2.0 μm square pixels. Data files were up to 60 Mb long and exposure times ranged to several hours. No sidewall etch corrections were applied to the pattern and proximity effects were only treated approximately. A Fresnel phase lens was fabricated that had diffraction limited optical performance with 83% efficiency.

I. INTRODUCTION

Surface contouring an e-beam resist by controlling both the exposure dose and the development process was demonstrated by Fujita[1] *et al.* in 1981. They designed, fabricated, and tested micro Fresnel-zone plates, blazed gratings, and Fresnel lenses in polymethyl methacrylate (PMMA). The exposure method used involved scanning the e-beam, in either straight lines or circles, with the dose adjusted to give the desired surface depth after development. This method produced somewhat irregular groove shapes, but efficiencies of 50%–60%, with near-diffraction-limited performance were achieved. More recently Ekberg[2] *et al.* reported on kinoform phase holograms. These were patterns comprising a 512×512 array of 10 μm square pixels, each with a unique e-beam exposure dose calculated to give the appropriate etch depth upon development. Ten doses/depths were used. Diffraction efficiencies of 70% were reported. In this paper, we report upon the fabrication, physical, and optical characterization of kinoforms comprising up to 3001×3001 arrays of 0.3–2.0 μm pixels that encode 16-level phase holograms. The effects of resist contrast, e-beam proximity dose, and sidewall etching are considered. Atomic force microscopy, channel fringe spectroscopy, scanning electron microscopy (SEM), and optical microscopy have all been used to characterize the fabricated devices. A Fresnel lens with 83% efficiency producing a diffraction limited focal spot has been fabricated.

II. EXPERIMENT

A. Resist characterization

High contrast (gamma) is a desirable property for photo- and e-beam resists used for device patterning. With high gamma, large variations in exposure dose will have little effect on the pattern shapes as long as the exposure is above a critical level. Hence, the common resists and their development processes have been tailored in this direction. In the present application, however, unity contrast is desired. The developability of the resist would, ideally, be linearly related to the exposure dose. The early work reported by Fujita[1] employed PMMA as the resist with development temperature controlled at 10 °C, leading to very high contrast—small changes in exposure led to large changes in developability and etch depth. This certainly contributed to the roughness observed in the etch profiles. The present work began with a search for an appropriate e-beam resist process having near-unity contrast.

The first system tried was a Rohm and Haas Company experimental acid hardening positive resist tailored to have low contrast (ECX-1151). Initial experimentation revealed that after partial development, the remaining material was highly inhomogeneous, leading to unacceptable optical scattering.

Earlier work with PMMA had revealed no such problem—partial development simply removed a uniform layer of surface material. However, as noted above, PMMA is normally a very high gamma material. It is known however that contrast is governed by the develop-

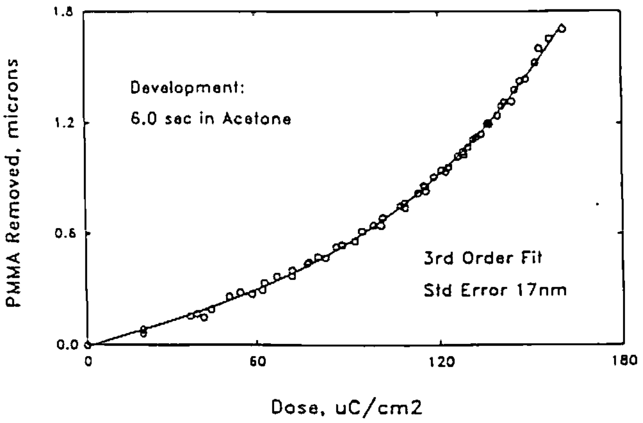

FIG. 1. PMMA exposure dose sensitivity using pure acetone as the developer at 21 °C. The data were fit by a third-order polynomial with a standard error of ±17 nm. This is equivalent to an optical phase shift, relative to air, of ±λ/75.

ment process and in particular that aggressive development of slightly under exposed resist leads to decreased gamma. In a series of tests, it was found that by developing partially exposed PMMA in pure acetone (its usual *solvent*), contrast could be markedly reduced. Development time decreased drastically, however, to roughly 5 s, and had to be controlled to ±0.1 s. This was accomplished using a Solitec resist spinner equipped with a Tridak resist dispense head. Acetone was introduced onto a spinning sample (500–3000 rpm) through the Tridak head for durations controlled by a computer to tenths of a second. The development process was terminated by a powerful blast of nitrogen gas, again controlled by computer. A series of shorter and shorter development steps was used to achieve precise etch depths. This avoided the need for careful temperature control. Figure 1 illustrates typical exposure versus development data. The principal method used to measure film thickness was channel fringe spectroscopy, wherein the interference fringes produced by light reflected from the PMMA top surface and from the substrate surface are spectroscopically measured and analyzed. This was accomplished using the Leitz MPV-SP instrument. The data of Fig. 1 were fit by a third-order polynomial, and that analytic expression was used to compute the dosage necessary to produce a desired etch depth. Further tests revealed that, within experimental error, etch depth was linearly related to development time.

The proximity effect—exposure dose contributed by scattered electrons—plays a very important role in the present work. Much study has been given to the effect in the literature. It is found that typically 30% of the exposure dose at the center of a large uniformly exposed field can arise from electrons backscattered from the substrate. For the present purposes, the spatial distribution of this proximity effect dose can be modeled as a Gaussian[3] of the form

$$D_p(r)/Q_0=\frac{\eta}{\pi\alpha^2}\exp(-r^2/\alpha^2),$$

where D_p is the proximity dose intensity at distance r from a primary point dose Q_0 delivered at $r=0$, η is the proximity factor, and α is the range of the Gaussian. Both η and α depend strongly on substrate composition and geometry and upon the electron beam voltage. α is typically 2–5 μm. In the current work, we seek to control the absolute resist thickness to better than 60 nm and the relative thickness from pixel to pixel to better than 20 nm. This requires dose control at the percent level. Clearly, proximity effects must be taken into account. In the work reported here, it was assumed that all pattern variations averaged out over the proximity range. This means that at each pixel, the proximity dose would be the same, and equal to that caused by the average primary dose. For the Fresnel lens patterns used in the current work, this approximation fails near their center (in the first few Fresnel zones) where the etch depth changes only slowly with distance. It is not a good approximation at all when the pixel size itself approaches the proximity range. Effects due to this error are clearly visible in the results and probably account for much of the observed loss in optical efficiency.

It should be noted that an exact solution for the proximity effect is possible in the present situation, which is not the case usually. In the usual application of e-beam lithographic practice, one desires that the resist be either fully developed or totally undeveloped. Since at the boundary of exposed areas, the proximity effect will always lead to exposure of the adjacent region, and negative dose is not possible, no exact solution is achievable. In the present case, every point in the pattern receives a finite primary dose (that might even include a bias value introduced to enable the correction). These doses can be adjusted both up and down to account for the proximity dose delivered from surrounding pixels. Several mathematical schemes are available to handle this correction. Deconvolution by Fourier transform seems appropriate, and work is in progress in this area.

B. Optical patterns

Two sets of patterns have been fabricated. The first set comprised four patterns designed by F. Coetzee and D. Casasent at the Center of Excellence for Optical Data Processing, Carnegie Mellon University. These described two astigmatic Fresnel lenses using up to 6000×1861 pixels 0.3 μm square, a linear array of 17 cylindrical lenses with 1000×4250 1.0 μm square pixels (all for refocusing the output beams of near-IR solid state diode lasers), and an array of superimposed cylindrical lenses, each with a different orientation and linear phase term with 4000×4000 2.0 μm square pixels (to perform an optical Hough transform). The data was in the form of pixel by pixel phase delay, rounded to the nearest 1/16th of a wavelength with error diffusion. Code was written to process this information directly into the "J51" format needed to drive the pattern generator of the JEOL JBX-5DII e-beam writer. The code grouped adjacent pixels having the same phase delay into single patterns. This afforded significant data compression, particularly for the cylindrical lenses. The code also resized each pattern to eliminate overlap at boundaries. The first three patterns were exposed, developed and physically characterized, but no data is available on their optical performance. The Hough transform pattern, larger to begin with and much less compressible, was too large (> 250 Mb) to handle in a single exposure and it was not fabricated.

A Fresnel phase lens was then designed at Jet Propulsion Laboratory (JPL) for use at the wavelength of the helium neon laser where it could be tested. To achieve a 360° phase shift, in PMMA, relative to air, requires a thickness of $\lambda/(n-1)=1.29$ μm. Pixels at the Fresnel-zone boundaries will differ in etch depth by that amount. Away from the boundaries, the difference in thickness of adjacent phase delay zones will be 1/16 of that, or 0.08 μm. Due to the isotropic nature of the development process, as a step is exposed, its lower riser will begin to etch laterally at the same rate that the step land etches down. This implies that [illegible] risers will always be tilted back at an angle whose tangent is given by the ratio of the etch rates of the adjacent steps. For the two deepest steps, that ratio approaches one, and the riser angle approaches 45°. At the Fresnel-zone boundaries, the step edge will recede laterally at the same rate that it descends. For the base dose chosen, 20 μC/cm^2, that amount at full development was approximately 0.1 μm. These sidewall etching effects cannot be avoided, but their influence on the optical performance could be minimized by adjusting the pixel shapes in the data pattern. This was not attempted in the present work, but to minimize their importance, a 1.0 μm pixel size was chosen. Data set size and exposure time constraints dictated total pattern area. A square 3001×3001 array was chosen. The requirement of 16 phase levels in the outermost full circle zone determined the design focal length, 38 mm. Data was processed as described above, yielding an exposure file 51 Mb long. Total data processing time was 1 h, and the e-beam exposure time was 90 min. This lens has [illegible]en fabricated and fully characterized both physically and optically.

C. Fabrication details

The JPL-designed Fresnel lens described above was exposed using the JEOL JBX-5DII e-beam lithography system at the Center for Space Microelectronics Technology Microdevices Laboratory. A beam current of 3.0 na, beam waist diameter ~0.3 μm, and step size of 0.2 μm were used. The minimum dose was set at 20 μC/cm^2. Less than this produced so little solubility increase that unexposed PMMA was removed too rapidly during development. At the chosen dose, the dissolution rate ratio of unexposed to minimally exposed PMMA was an acceptable 4:1. The shot time for these conditions was 2.7 μs. Shot time for the JEOL JBX-5DII is quantized in increments of 50 ns. This, together with the nonlinearity of the dose-response curve for PMMA, sets a limit on the upper dose that can be used—at high dose, the difference in exposure time needed to produce a 1/16 wavelength phase step decreases, ultimately approaching the quantization limit. For the stated conditions, the difference in shot time between the most deeply etched phase steps was 13 clock ticks enabling the step size to be set within 8%. The doses needed to produce 16 equally spaced etch depths were determined using data like that shown in Fig. 1. The approximation was made that the proximity dose at each step would be the same and equal to that produced by the median primary dose. As noted above, this approximation is inadequate for the central Fresnel zones.

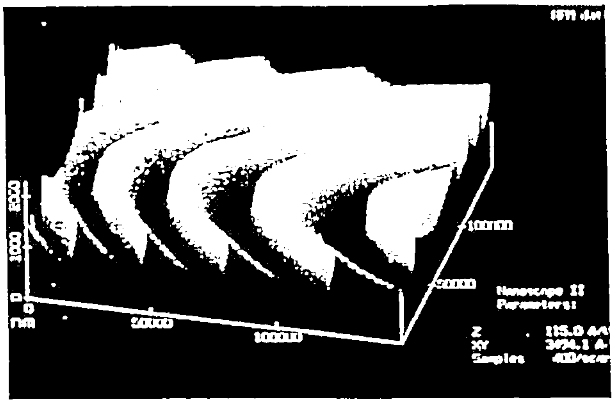

FIG. 2. AFM topographic image of a section of an astigmatic Fresnel phase lens rendered in 16 phase steps in PMMA by e-beam lithography.

The substrates were 1/8 in. thick, 1 in. diam 1/10 wave optical flats of BK7 glass. These were first prepared with gold fiducial marks so that the e-beam could be focused, and its deflection factors calibrated, directly at the exposure plane. A 2 μm thick layer of PMMA was built up on the surface by 4 applications of 950 K molecular weight polymer in 5% solution in chlorobenzene spun at 3000 rpm. Thorough baking (170 °C, 60 min) between applications produced a uniform film—there was no evidence of vertical inhomogenity in the final results. A 100 Å layer of aluminum was applied over the PMMA prior to exposure to dissipate charge. This was stripped in mild alkali prior to development which proceeded as described above. Additionally, sixteen 50×50 μm test patches were exposed, with doses calculated to give equally spaced etch depths, that could be used to monitor the progress of the development. To facilitate that measurement, the test patches were exposed over a region of the substrate that was coated with 200 Å of aluminum prior to spinning on the resist film. Without the aluminum, the channel fringes used to measure the film thickness had near-zero visibility because the refractive indices of PMMA glass are nearly equal (1.49 versus 1.54).

III. RESULTS

A. Physical characterization

Figure 2 is a three-dimensional representation of an area near the center of one of the astigmatic Fresnel lenses, produced by a scanning atomic force microscope [Digital Instruments Nanoscope atomic force microscope (AFM)].

No evidence of field stitching or pattern overlap can be discerned. Even small errors of this sort produce dramatic effects as etch depth will be doubled in areas of pattern overlap. Individual phase steps can be seen. Note that the steepness of the vertical back walls is much enhanced in the figure because of the difference in vertical and horizontal scales. In fact, the steepness recorded by the AFM is limited by its tip geometry. The instrument used to acquire the data for Fig. 2 had is a pyramidal cone with an apex angle of ~114°. Scanning electron microscopy (SEM) data and AFM data taken with ultra sharp tips, indicate that back wall steepness exceeded 60°. High resolution AFM topographic data indicate that the surface roughness of the partially developed PMMA was of the order of ±5 nm. Quantitative AFM profile data reveal that in the central Fresnel zones, too much PMMA has been removed in the deeply etched regions, and too little in the shallow regions. This is due to the failure of our approximate treatment of the proximity effect. Similar data taken at the pattern edges where the Fresnel zones are narrower than the proximity range show the desired linear ramp.

Physical examination of the JPL-designed Fresnel lens showed a similar result. Optical film thickness data was taken at the center of the pattern where the individual phase plateaus were broad enough to permit measurement with the Leitz MPV-SP instrument. Again, the effects of inadequately treating the proximity effect were apparent.

B. Optical characterization

A knife-edge test was performed to access the optical performance of the JPL-designed Fresnel phase zone lens. An expanded, collimated helium neon laser beam was focused by the lens, and a razor-blade knife edge was mechanically driven across the focal point, in the focal plane. Energy passing the knife edge was monitored by a photodiode detector. It was found that 83% of the incident light energy was focused (first diffraction order), 14% was redirected into high diffraction orders, and 3% passed through the lens undeviated (zeroth order). An attempt was made to adjust the final etch depth to minimize the zeroth order energy. Figure 3 shows the intensity of the first order radiation as a function of knife-edge position, plotted together with a curve derived by integrating the Airy function that indicates diffraction limited performance. The data points fit the predicted curve within experimental limits. This result might be anticipated on the basis that the patterning precision of JEOL JBX-5DII lithography tool, ±50 nm, is essentially perfect on the scale of the Fresnel-zone pitch and diameter. The high-order radiation formed a set of concentric circles in the far field. This would be predicted for energy diffracted by an error in the phase step profile that repeated in each Fresnel zone. Its origin can therefore be tentatively ascribed to the known errors in profile introduced by our inadequate treatment of the proximity effect.

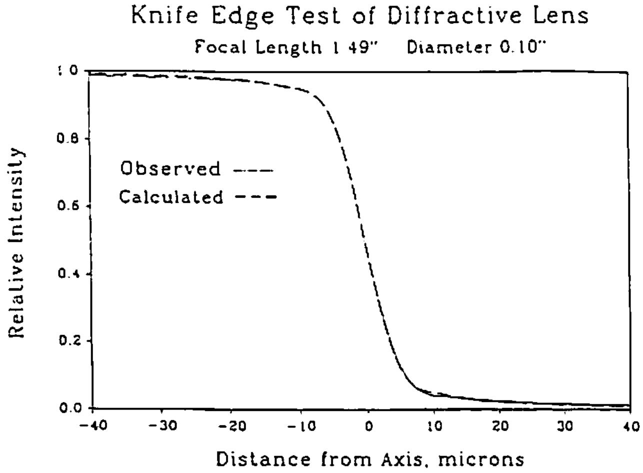

FIG. 3. Observed energy passed by a knife-edge scanned in the focal plane of the JPL-designed Fresnel phase lens. The calculated curve represents a diffraction limited focus. 83% of the energy incident on the lens passed through the focal spot, 14% was diffracted into higher orders, and 3% was unfocused.

IV. CONCLUSIONS

The results obtained indicate that direct write, partial exposure e-beam lithography can produce excellent transmissive optical elements for use in the visible. It is anticipated that a more careful treatment of proximity effects will result in devices with >90% efficiency. Aspect ratio and sidewall etching issues need study before pixel size can be reduced and focal lengths shortened. Work is in progress in these areas.

ACKNOWLEDGMENTS

The research described in this article was performed by the Center for Space Microelectronics Technology, Jet Propulsion Laboratory, California Institute of Technology, and was jointly sponsored by the Strategic Defense Initiative Organization/Innovative Science and Technology Office and the National Aeronautics and Space Administration/Office of Aeronautics and Space Technology. The authors wish to thank Karen Graziano, Rohm and Haas Company, for supplying a sample of ECX-1151 together with process data.

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