N94-17348

PHASE HOLOGRAMS IN PMMA WITH PROXIMITY EFFECT CORRECTION

P.D. Maker and R.E. Muller
Center for Space Microelectronics Technology, Jet Propulsion Laboratory
California Institute of Technology, Pasadena, CA 91109

ABSTRACT

Complex computer generated phase holograms (CGPH's) have been fabricated in PMMA by partial e-beam exposure and subsequent partial development. The CGPH was encoded as a sequence of phase delay pixels and written by the JEOL JBX-5D2 E-beam lithography system, a different dose being assigned to each value of phase delay. Following carefully controlled partial development, the pattern appeared 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 $\pm\lambda_{vis}/60$. 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 CGPH's with pixel size of the order of the e-beam proximity effect radius, the patterns must be corrected for the extra exposure caused by electrons scattered back up out of the substrate. This has been accomplished by deconvolving the two-dimensional dose deposition function with the desired dose pattern. The deposition function, which plays much the same role as an instrument response function, was carefully measured under the exact conditions used to expose the samples. The devices fabricated were designed with 16 equal phase steps per retardation cycle, were up to 1 cm square, and consisted of up to 100 million 0.3-2.0 μm square pixels. Data files were up to 500 MB long and exposure times ranged to tens of hours. A Fresnel phase lens was fabricated that had diffraction limited optical performance with better than 85% 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 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% to 60%, with near-diffraction-limited performance were achieved. More recently Ekberg[2] et. al. reported on kinoform phase holograms. These were patterns comprising a 512x512 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 an earlier paper[3], we reported upon the fabrication, physical, and optical characterization of kinoforms that encoded 16-level phase holograms and a Fresnel lens having a diffraction limited focal spot and 83% efficiency. At that time, only an approximate treatment of the E-beam proximity effect was used. The fabrication imperfections thus introduced caused significant performance degradation. In this paper, we report upon a technique for treating the proximity effect exactly and upon the performance of an off-axis Fresnel lens fabricated using the method. The lens had well shaped grooves with none of the peak-rounding and valley-filling that characterize the proximity problem, and its far-field pattern was very uniform. Near-diffraction limited focal spots were measured, and its overall efficiency was ~86%.

II. EXPERIMENT

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 development 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 5-10 sec, and had to be controlled to ± 0.1 sec. This was accomplished using a Solitec resist spinner equipped with a

Tridak resist dispense head. Acetone was introduced onto a spinning sample (1000 rpm) through the Tridak head for durations controlled by a computer to tenths of a second. The development process was terminated abruptly 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 vs. development data.

Initially, film thickness was measured by channel fringe spectroscopy, wherein the interference fringes produced by light reflected from the top and bottom surfaces of a film are spectroscopically measured and analyzed. This was accomplished using the Leitz MPV-SP instrument. To measure PMMA films on glass, however, it was found necessary to deposit a thin film of aluminum on the substrate. Because the index of refraction of PMMA nearly matches that of glass, no usable interference fringes are produced by that interface. Etch depth data like that shown in Figure 1 were fit by a third-order polynomial, and that analytic expression was used to compute the dosage necessary to produce any desired etch depth. Further tests revealed that, within experimental error, etch depth was linearly related to development time.

A Digital Instruments Nanoscope III Scanning Probe Microscope (SPM) has recently been installed. Surface profiles at sub-wavelength resolution, as well as film etch depth, are conveniently measure using it. Further, actual devices can be examined directly as the SPM tip scans them without damage.

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 back-scattered from the substrate. For the present purposes, the spatial distribution of this proximity effect dose can be modeled as a Gaussian[4] of the form

$$D_{prox}(\vec{r}) / Q_o = \frac{\eta}{\pi\alpha^2} \exp(-\vec{r}^2/\alpha^2)$$

where D_{prox} is the proximity dose intensity at distance r from a primary point dose Q_o delivered at r = 0, η is the proximity factor, and α is the range of the Gaussian. The total dose arriving at point r due to a spatially varying, patterned primary dose D_{prim}, can be expressed as a convolution of an effective point spread function with that patterned dose, as:

$$D_{tot}(\vec{r}) = \iint d(\vec{r} - \vec{r}_o) \times D_{prim}(\vec{r}_o) \times PSF(\vec{r} - \vec{r}_o)$$

$$= D(\vec{r}) \otimes PSF(\vec{r})$$

where

$$PSF(\vec{r}) = \delta(\vec{r}) + \frac{\eta}{\pi\alpha^2} \exp(-\vec{r}^2/\alpha^2)$$

Both η and α depend strongly on substrate composition and geometry and upon the electron beam voltage. α is typically 2 to 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 an earlier paper[3] we reported on fabrication of Fresnel lens which treated the proximity dose problem only approximately. That lens displayed groove shapes that had rounded off tops and filled in trenches. It was at first suspected that the development procedure caused the problem. A series of experiments employing significantly different techniques convinced us that development was not causing the problem. The proximity effect can also cause such distortion in the groove shapes. At zone boundaries excess proximity dose from the heavily exposed valley regions overlaps into the lightly dosed areas, and vice-versa. In the present work, the proximity effect has been carefully measured, and corrections for it have been applied to the pattern.

Having calibrated the e-beam dose sensitivity of PMMA, direct measurement of proximity parameters becomes possible. A square area many times larger than the range, α, of the effect is first exposed with a measured, heavy dose. After controlled development, the PMMA at the perimeter of the area is seen to be thinned in a characteristic way by the proximity dose. Integrating the above expression for geometry leads to the following expression for the decay of the proximity dose with distance from the edge of the exposed area:

$$D_{prox}(x) = \frac{\eta}{2} \times D_{prim} \times \mathrm{erfc}(x/\alpha)$$

where erfc(x) is the complimentary error function. Fig. 2 shows a fit of such data using the parameters $\alpha = 5.25$ microns and $\eta = 0.25$.

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 represents a straightforward approach. If $\mathbf{P}(\mathbf{r})$ is the desired exposure pattern and $\boldsymbol{P}(\mathbf{k})$ its Fourier transform, and $\mathbf{PSF}(\mathbf{r})$ is the point spread function and $\boldsymbol{PSF}(\mathbf{k})$ its Fourier transform, then $\boldsymbol{Pc}(\mathbf{k})$, the Fourier transform of the desired function is:

$$\boldsymbol{Pc}(\mathbf{k}) = \boldsymbol{P}(\mathbf{k}) / \boldsymbol{PSF}(\mathbf{k}).$$

$\mathbf{Pc}(\mathbf{r})$ is then obtained by inverse transforming $\boldsymbol{Pc}(\mathbf{k})$. A carefully optimized two dimensional fast Fourier transform deconvolution program has been implemented on our VAX Station 3100 computer. It handles a 4K by 4K problem in five hours.

Indeed, it was found necessary to recess typical patterns by ~0.2 microns with bias dose to eliminate negative dose.

Consideration of the isotropic nature of the development/etching process being use here reveals that the risers separating adjacent lands etched to near-identical depths will be sloped at 45 degrees. Further, a shallow land adjacent to a very deep one will be etched laterally by an amount equal to its depth. Worse, a land half as deep as its neighbor will be etched laterally by half its depth. Noting that a transmission phase plate for use at the wavelength of the red helium neon laser must have groove depths of $\lambda/(n-1) = 1.29$ microns, it becomes apparent that the pixel size of kinoform-encoded CHG's must be larger than about one micron to avoid serious degradation at the hands of isotropic etching. Note that for patterns resembling uniform, curved gratings, as do those for simple lenses, the case of half-height risers, which leads to maximum lateral etching, does not arise as long as there are many pixels in the narrowest Fresnel zone.

The JEOL JBX-5D2 EBLS pattern data format requires sixteen bytes to specify a single elementary pattern unit, and cannot handle files longer than 512 MB. Thus a kinoform-encoded CGH can be no larger than 5K by 5K. To uniformly expose a 1 micron square pixel, an e-beam spot size of ~0.2 micron diameter should be used and it should increment between exposure doses by less than about 0.1 microns. The JBX-5D2 also has a maximum shutter speed of 0.5 microseconds. Since an area 0.1x0.1 microns square is exposed at each click, and a minimum dose of $20 \mu C/cm^2$ is expected, the beam current must be limited to less than 3 na. Under these exposure conditions, one square cm can be exposed in ~15 hours.

Based upon the above considerations, an exposure pattern for an off-axis Fresnel lens 3 mm square, with 3751x3751 square pixels each 0.8 micron on a side, having a focal length of 38 millimeters, and with its center of curvature 2 mm from the lens center, was prepared. First, the requisit phase delay was calculated as a floating point number for each pixel in a 4K by 4K array. These numbers were converted first to equivlent PMMA thickness to be removed and then to total electron dose, this based upon nonlinear calibration data like that show in Fig. 1. Next, the Fourier deconvolution proceedure was appled. The corrected dose pattern was then cropped to its central 3751 rows and columns eliminating wrap-around errors introduced by the FFT technique. A histogram of all the doses was assembled, and divided into 64 bins, equal numbers of pixels in each bin. This data set was then encoded in the format used by the JEOL EBLS. An algorithm was used that grouped adjacent pixels with identical exposure into larger rectangular patterns, thus achieving modest data compression. In place on the PDP 11/84 that controls the EBLS, this data set was some 125 MB long. Individual 3 mm square Fresnel lenses require 90 minutes to expose. Development time in pure acetone was 11 seconds.

This and like lenses have been fabricated and fully characterized both physically and optically. They were etched in 2 micron thick PMMA films on 1/10 wave BK7 glass optical flats. The PMMA was built up by 4 applications of 950K 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.

III. RESULTS

A. Physical Characterization

Figure 3 is a three-dimensional representation, produced by the SPM, of an area near the center of a characteristic Fresnel lenses. 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 SPM is limited by its tip geometry. The instrument used to acquire the data for Figure 3 had is a pyramidal cone with an apex angle of ~114 degrees. SEM data and SPM data taken with ultra sharp tips, indicate that back wall steepness exceeded 60 deg. High resolution SPM topographic data indicate that the surface roughness of the partially developed PMMA was of the order of ±10 nm. Quantitative SPM profile data reveal flat ramps extending from valley to ridge with no sign of the rounding that plagued the earlier lenses.

B. Optical Characterization

A knife-edge test was performed to access the optical performance of the JPL-designed Fresnel 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 photo diode detector. It was found that 86% of the incident light energy was focused (first diffraction order), <2% was redirected into high focusing orders, <1% was diffracted by the 'pixel grating', 1.6% passed through the lens undeviated (zeroth order), and about 10% of the incident radiation was scattered diffusely. An attempt was made to adjust the final etch depth to minimize the zeroth order energy. Figure 4 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.

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. Efforts are underway to reduce the diffuse scattering,, and it should be possible to produce devices with > 90% efficiency. Aspect ratio and side wall etching issues need study before pixel size can be reduced and focal lengths shortened. With the patterning positioning accuracy available, it is possible to overlay these phase patterns on a transmission mask, producing CGH's with controlled amplitude as well as phase. Work is also in progress in all of these areas.

ACKNOWLEDGMENTS

The research described in this paper 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.

FIGURE CAPTIONS

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 $\pm\lambda/75$.

Fig. 2. Fit of the experimentally measured proximity dose at the edge of a heavily exposed half-plane to the complementary error function.

Fig. 3. AFM topographic image of a section of a typical Fresnel phase lens rendered in 16 phase steps in PMMA by E-Beam lithography.

Fig. 4. 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.

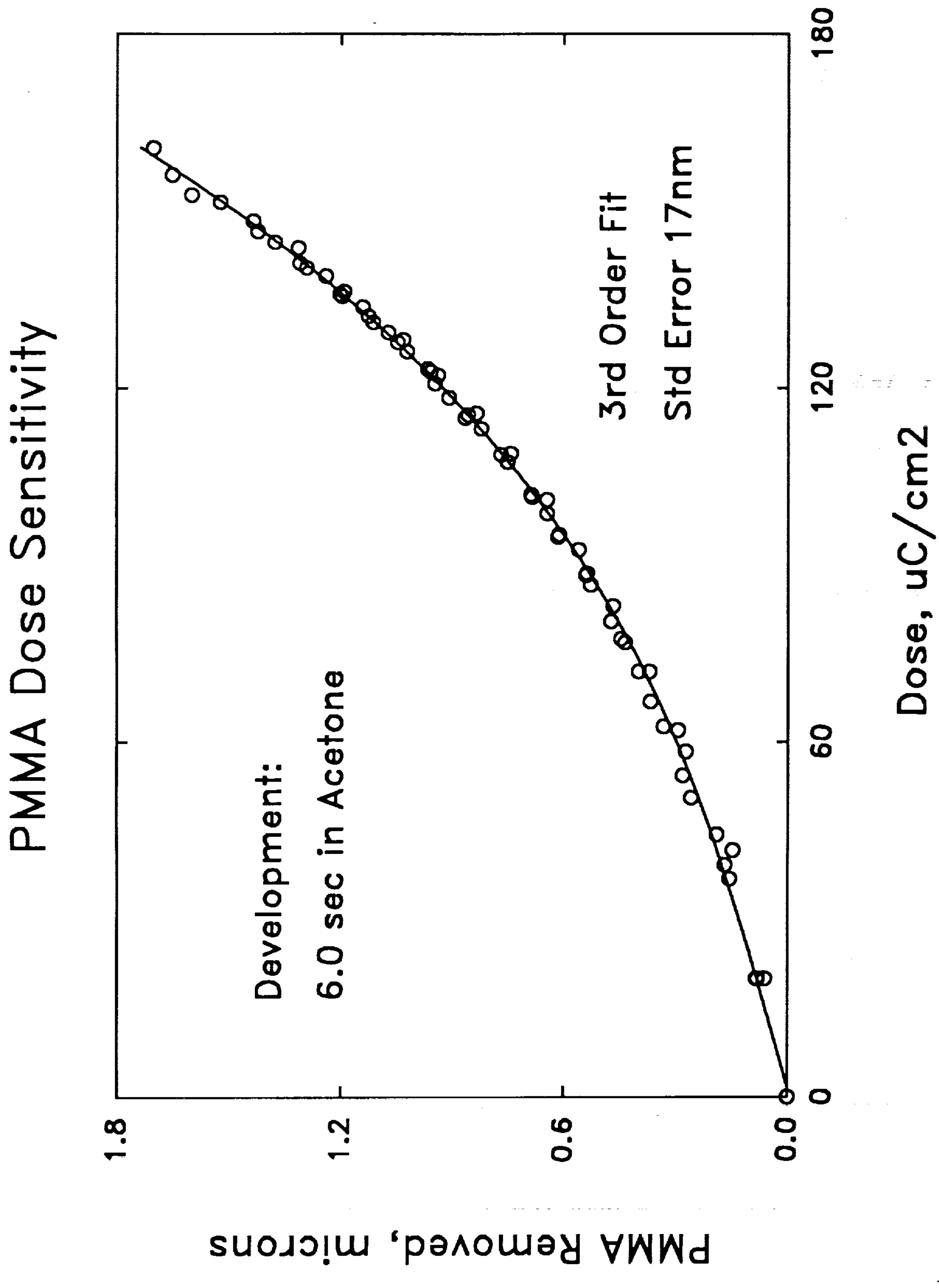
REFERENCES

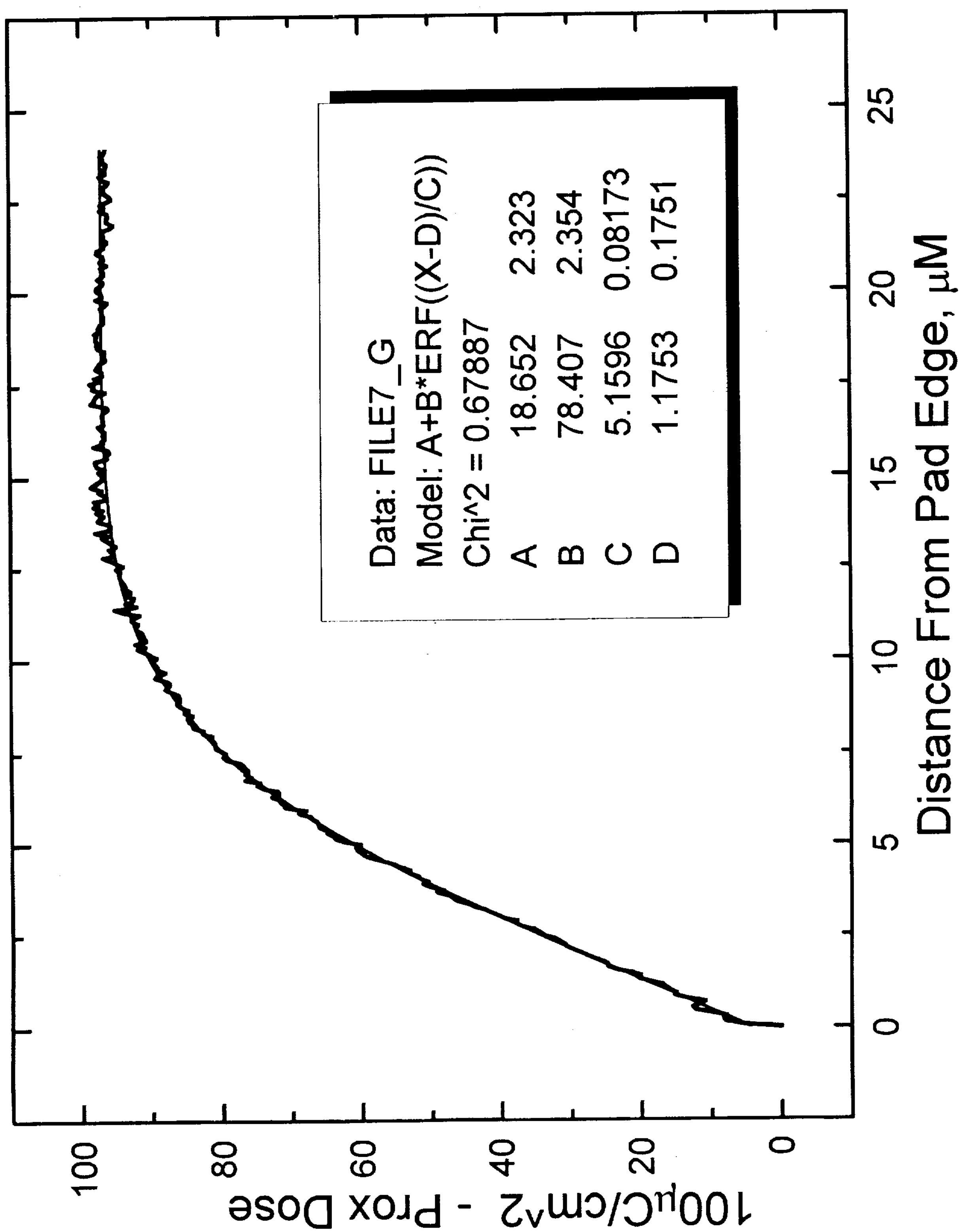
1 T. Fujita, H. Nishihara, and J. Koyama, Opt. Lett. **6**, 613 (1981). T. Fujita, H. Nishihara, and J. Koyama, Opt. Lett. **7**, 578 (1982).

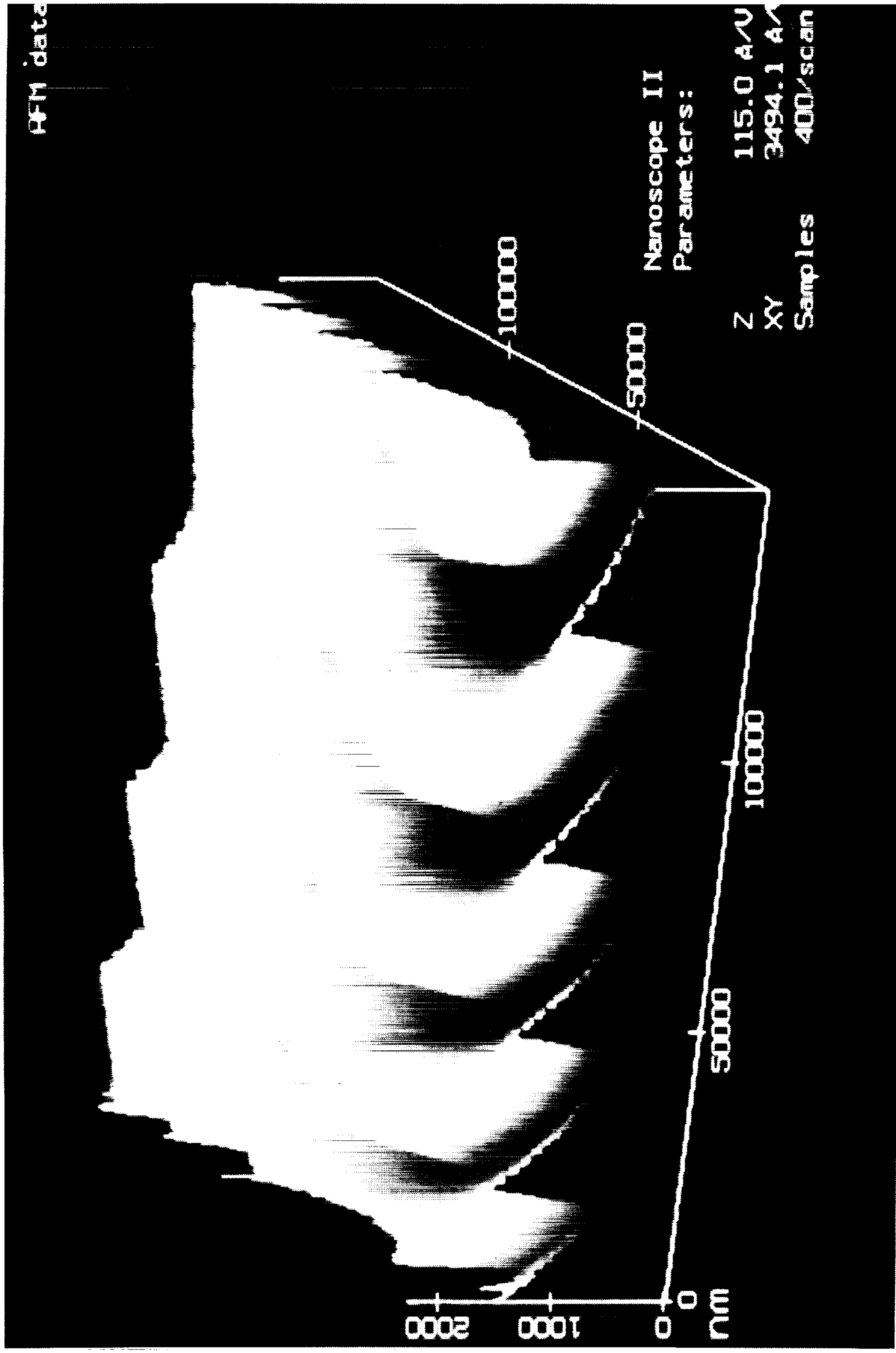
2 M. Ekberg, M. Larsson, S. Hård, and B. Nilsson, Opt. Lett. **15**, 568 (1990).

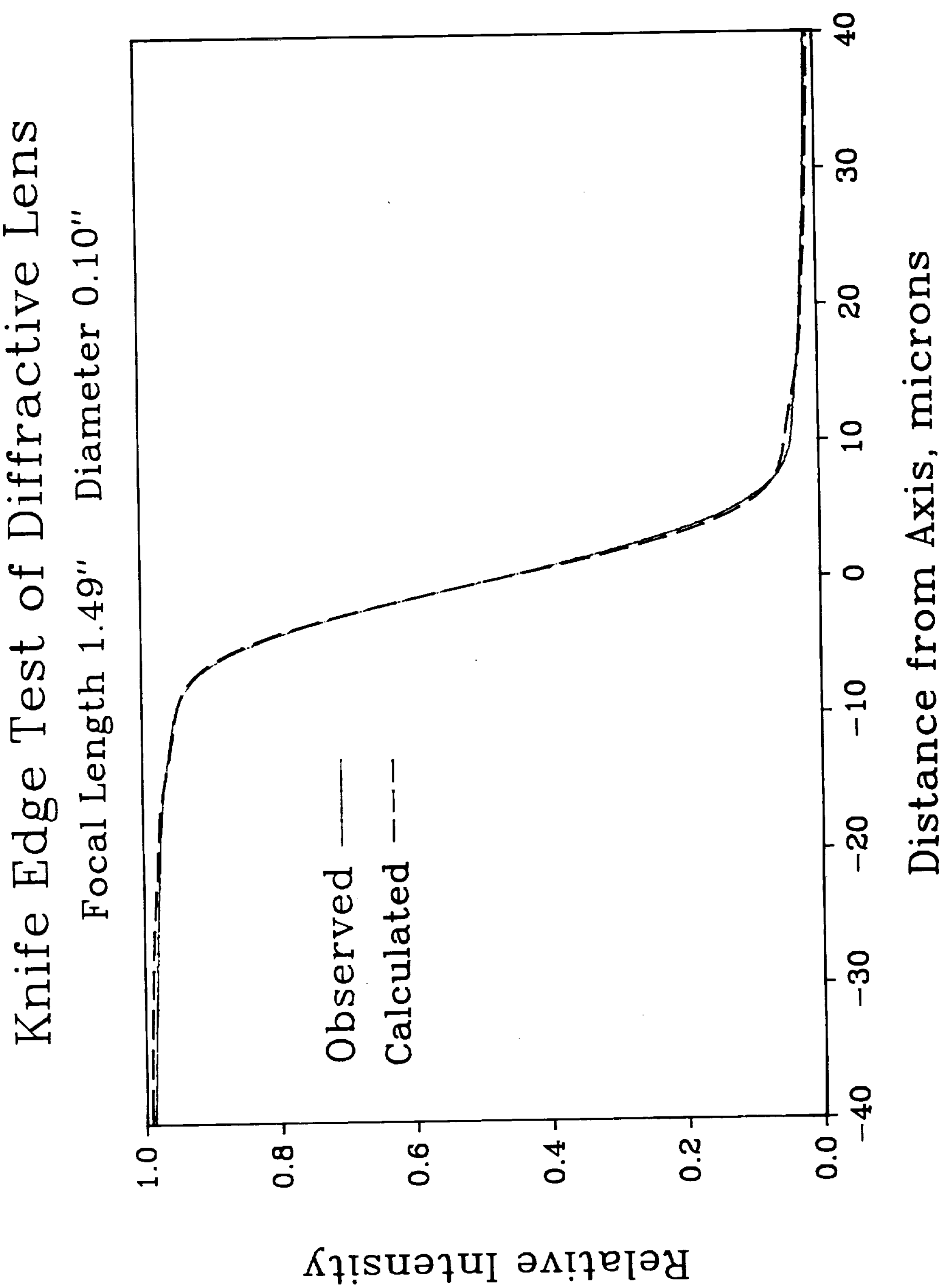
3 P.D. Maker, and R.E. Muller, J. Vac. Sci. Technol. B **10**, 2516 (1992)

4 M. Gentili, L. Grella, A. Lucchesini, L. Luciana, L. Mastrogiacomo, and P. Musumeci, J. Vac. Sci. Technol. B **8**, 1867 (1990)


PMMA Dose Sensitivity
Development:
6.0 sec in Acetone
3rd Order Fit
Std Error 17nm
0
60
120
180
Dose, uC/cm2
0.0
0.6
1.2
1.8
PMMA Removed, microns


Data: FILE7_G
Model: A+B*ERF((X-D)/C))
Chi^2 = 0.67887
A 18.652 2.323
B 78.407 2.354
C 5.1596 0.08173
D 1.1753 0.1751
Distance From Pad Edge, μM
100μC/cm^2 - Prox Dose
0
5
10
15
20
25
0
20
40
60
80
100


AFM data
Nanoscope II
Parameters:
Z 115.0 Å/V
XY 3494.1 Å/
Samples 400/scan
100000
50000
100000
50000
0
2000
1000
0
nm



Knife Edge Test of Diffractive Lens
Focal Length 1.49"   Diameter 0.10"
Observed
Calculated
Relative Intensity
0.0
0.2
0.4
0.6
0.8
1.0
−40
−30
−20
−10
0
10
20
30
40
Distance from Axis, microns