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Fabrication and Characterization of III-V Compound Semiconductor Bragg-Fresnel Lenses for Hard X-Ray Microfocusing

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Hard x-ray phase Bragg-Fresnel lenses (BFLs) have been made in III-V semiconductors of (111) GaAs and InP, and in Si for comparison purposes. Diffractive linear and circular patterns were defined with conventional electron beam lithography. Pattern transfer was accomplished with reactive ion etching (RIE) using gases of $BCl_3/Cl_2/SiCl_4$, $CH_4/H_2/Ar$, and BCl_3/Cl_2, respectively. In addition, selective etching of heteroepitaxial GaAs devices for better process depth control was demonstrated by incorporating an epitaxial AlGaAs etch stop layer between the GaAs top layer and substrate and then etching with Cl_2/O_2. Preliminary tests on focusing have been made at the Stanford Synchrotron Radiation Laboratory (SSRL) showing the ability to focus a 50 micron x-ray beam to ~5 microns for Si structures. This is an essential requirement for x-ray microprobe technology development which has wide applications in x-ray imaging, deep field x-ray lithography, and in probing nanometer scale complex fluids such as boundary lubrication layers.

1. INTRODUCTION

The design and fabrication of high performance optics that will focus hard X-rays of wavelength ~ 1Å can augment our ability to perform structural studies of complex material and biological systems with high spatial resolution and under confined geometries. Bragg-Fresnel -Lenses (BFLs), which are Fresnel zones etched into the surface of substrates, have been shown to focus hard X-rays at energies up to 100 keV [1] with efficiencies of 40%, close to the theoretically predicted performance. The primary material used for the BFLs have thus far been Si and Ge [2]. Requirements on the etch-transfer process for the BFLs are stringent: high lateral pattern fidelity, straight and smooth etched sidewalls and a precisely defined etch depth that is uniform over the BFL. The etched depth is determined by the required phase shift for X-rays traversing adjacent zones, and is usually several microns for Si BFLs [2]. The depth is inversely proportional to the density of the substrate material, and the choice of higher density compound semiconductors, such as GaAs and InP will require more shallow etched depths. This can prove to be a critical advantage in the fabrication of the BFLs, exacting fewer demands on the durability of the masking material or the stability of the etch process over long periods of time. Moreover, the widespread availability of heterostructure materials in these compound semiconductors (e.g. GaAs/AlAs) allows us to incorporate highly efficient etch-stop layers into the structures, ensuring the uniformity of the etched depth in these BFLs. We report here on our initial fabrication of GaAs and InP BFLs, and the preliminary evaluation of Si and GaAs-based BFLs.

2. FABRICATION

2.1 Pattern generation and e-beam lithography

The BFLs in this study were either 300 μm square (linear) or 300 μm diameter (circular) consisting of 150 zones with zone widths varying from 12.2 microns in the center to 0.5 microns at the outermost zone. Smaller arrays of 100 μm and 50 μm were also made. The patterns were generated on CAD tools using 0.025 μm pixel size; this corresponded to a 409.6 μm electron beam exposure field. Sixty-four-sided polygons were used to approximate circles and rings. Built-in software proximity corrections were applied to the linear structures, while circular zone plates were best realized without the corrections. The electron beam lithography tool is an IBM vector scan system upgraded with an FEI Schottky thermal field emitter gun operating at 25 keV [3]. All patterns were exposed within a single 409.6 μm field so that no field stitching was necessary.

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The bilayer (hi/lo) molecular weight PMMA resist scheme of Rooks et al. [4] was used to obtain metal mask lift-off of ~ 400-600Å of Ni. By employing a thicker bottom resist layer, up to ~1300Å of metal could be used. Above this metal thickness, the nickel would peel due to stress. Base resist dosages were 90-200 μC/cm^2, depending on the substrate material, and whether or not the proximity corrections were used.

2.2 RIE

All Reactive Ion Etching was carried out in various parallel plate systems having dedicated chemistries (e.g. chlorine for GaAs, methane/hydrogen for InP) with a 13.56 MHz rf excitation. Pumping was either with a diffusion or turbo pump, with ultimate pressures less than 10^{-6} Torr. Only the InP system did not have a sample loadlock. All systems were equipped with a He-Ne laser interferometry system for depth monitoring, and used where applicable. Etched depths were determined with a commercial stylus profilometer. Table 1 summarizes the etch systems and conditions used. A primary challenge for the etching of these BFLs is the uniform lateral and vertical etch required for a geometry where the line densities vary continually. Such a geometry is likely to aggravate microloading effects, in this case, a local depletion of the etching species for the denser areas of the pattern . Therefore although inspection of the etched GaAs BFLs at low magnification reveals smooth etching, faithful pattern transfer (the polygon approximation for the rings are in fact apparent) , and straight sidewalls (Figure 1), a closer inspection (Figure 2) shows that the denser regions of the linear GaAs BFL have not been cleared out completely. Similar effects pertain to the InP BFLs. The microloading, and the subsequent differences in etch rates leading to non-uniformity of etched depth, highlights the usefulness of a scheme that incorporates an etch stop layer. An effective etch stop layer allows overetching until all portions of the BFL have been satisfactorily etched. Our preliminary studies using an etch-stop structure utilizes heterolayers grown by MBE on a (111)B GaAs substrates. The growth started with a 5000Å GaAs buffer layer, followed by 1000Å $Al_{.7}Ga_{.3}As$, and thin 20Å AlAs layer, followed by GaAs

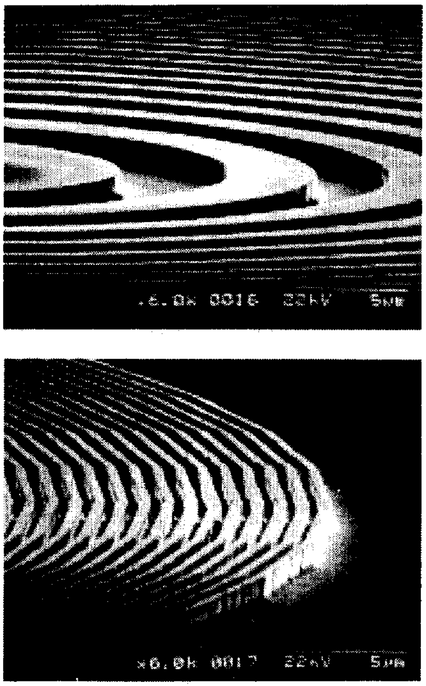

Figure 1. Bulk GaAs circular BFL showing central zones (top) and outer (bottom).

BFL Material	Gas Chemistry	Etch Rate (microns/min)	Flow-Pressure (sccm-mT)	Constant Power (W) or Voltage (V)	Desired Etch Depth: linear-circular (microns)	Comment
Si	Cl_2	0.1	20-5	200W	1.26 - 3.78	1, 2, 4
GaAs	$BCl_3/Cl_2/SiCl_4$	0.1	25/0.8/5-10	100W	0.53 - 2.12	1, 3, 6
InP	$CH_4/H_2/Ar$	0.1	4/20/10-75	500V	0.57 - 2.28	1, 3
GaAs/AlGaAs Heterostructure	Cl_2/O_2	0.2	7.5/1.0-1.5	350V	0.53 - 2.12	3, 5

Table 1 – RIE Systems and Conditions

1) Bulk Material, 2) Depth adjusted for (333) plane reflection, 3) Depth adjusted for (444) plane reflection, 4) BCl_3 added initially for oxide removal, 5) Only linear arrays attempted at this time, 6) Etch rate approximately 10% lower for (100) orientation

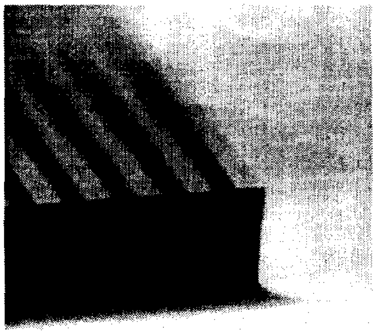

Figure 2. Bulk GaAs linear BFL showing microloading effects.

grown to the thickness of the desired BFL zone depth of 5300Å. The AlGaAs/AlAs comprise the etch-stop layer: a number of chemistries have been used to achieve etching of GaAs, selectively over AlGaAs. Our approach was to bleed a small amount of O_2 during the chlorine-based RIE of the heterostructure. The reactive Al will form a poorly-etchable Al-oxide layer through reaction with the introduced oxygen, thus slowing its etch rate with respect to that of the GaAs. The laser interferometer trace shown in Figure 3 demonstrates that the AlGaAs layer is indeed acting as an etch-stop. One period on the curve corresponds to 900Å of etched material, giving an etched depth of ~ 5400Å. Etching was continued for an additional 1.5 minutes after the laser signal had flattened out in order to fully 'clear out' the denser regions of the BFL. A segment of that BFL is shown in Figure 4 where it can be seen that the outer zone trenches have indeed been fully etched to the same depth as that of the open field area. Surface profiling showed an etched depth of 5700Å, which is larger than the expeected epi-layer thickness even after taking into account of the at most 3% deviation in growth thickness. This suggests that the slight etching had occured to the AlGaAs layer. Fine tuning of the growth/etching process parameters should allow us to precisely control the final etched depth to the desired value.

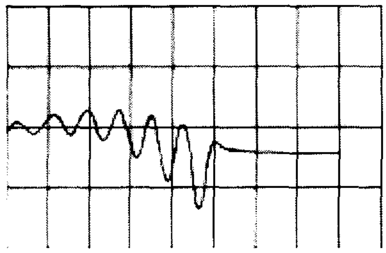

Figure 3. Laser interferometry signal on etched GaAs/AlGaAs heterostructure BFL.

Figure 4. Outer zone of etched GaAs/AlGaAs heterostructure.

3. X-RAY CHARACTERIZATION AND APPLICATIONS

X-ray characterization of linear BFLs fabricated on Si (111) and GaAs (111) substrates were conducted at Stanford Synchrotron Radiation Laboratory (SSRL) wiggler beamline 10-2. Experiments were done using 8 keV x-rays on 150 zone (total device size = 300 μm) BFLs as well as 25 zone (device size = 50 μm) BFLs (the length of zones are all 300 μm). Although focusing effects were observed to varying degree from all these devices, we found that the 25 zone (50 μm) device provided the cleanest focusing pattern with the smallest focal spot size (~ 5 μm) because the device size roughly matched the spatial coherence of the incident beam.

The experiment was set up as follows. The incident x-rays are defined by a set of X-Y slits (aperture) to match the size of the BFL which is located at 22 m from the x-ray source. The BFL was mounted on a translation/rotation stage assembly for precise positioning in the beam and for setting the Bragg angle. An aperture (~ 10 μm) mounted on a X-Y-Z stage was placed at the calculated focal distance of the BFL (~ 17 cm for the 50 μm BFL) followed by a NaI scintillation detector. The intensity distribution of the beam was characterized by scanning the pinhole in the focal plane. Such X-Y scans were performed at different distance (Z) from the BFL. Figure 5 shows a set of data taken from a 50 μm Si linear BFL. For comparison, the x-ray beam reflected from the Si substrate 150 μm above the BFL are presented. The focal spot size of ~ 5 μm is about an order of magnitude larger than the diffraction limited resolution of the BFL, indicating that the incident beam is only partially coherent. The diffraction limited resolution should be realized when the BFLs are utilized in a third generation synchrotron such as the Advanced Photon Source (APS) at Argonne.

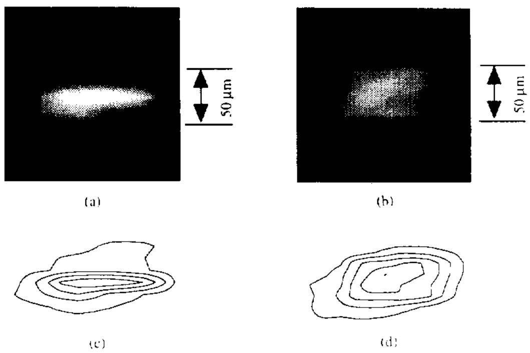

Figure 5. Images and contour plots of the x-ray beam with (a) (c) and without (b) (d) the BFL.

4. SUMMARY

We have presented a diffractive Bragg-Fresnel lens fabrication process that has successfully been implemented in GaAs and InP compound semiconductors. A GaAs/AlGaAs heterostructure incorporating an 'etch stop' layer was designed and processed. Our preliminary results indicate that the presence of the etch-stop layer significantly improves process latitude and mitigates etching non-uniformity, therefore warrants further study. At this point in time only the silicon devices have been tested for focusing hard x-rays, focusing a 50 micron x-ray beam down to ~5 microns. Experiments are underway to utilize the micron-size focused x-ray beam to probe the structures of complex materials and fluids under confined geometries (e.g. nanometer scale boundary lubrication layers) [5], and mesoscale complexes of DNA/protein and membrane forming lipids.

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