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Fabrication of small laterally patterned multiple quantum wells

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A technique of high voltage electron beam lithography and BCl_3/Ar reactive ion etching for laterally patterning $\text{GaAs}/\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ multiple quantum wells is described. The resulting structures were analyzed using scanning electron microscopy and a novel reflection electron microscopy technique, and their geometries are shown. Narrow columns 40 nm in diameter etched 230 nm through the quantum wells were reproducibly fabricated.

Microstructural control of GaAs and lattice matched AlGaAs by epitaxial growth techniques has enabled tailoring the electronic properties of these semiconductor materials. This has led to the design of new electronic and optoelectronic devices such as high electron mobility transistors and heterostructure lasers, and has revealed new physical phenomena such as the quantized Hall effect. Advances in technology for lateral patterning have motivated new research directions and device concepts to take advantage of geometrical confinement in more than one dimension.^{1,2} The degree of structural control obtainable in lateral patterning has not reached the nanometer dimensions attained by controlled growth, but high-resolution lateral patterning is improving and the range of geometries and materials increasing.^{3,4}

In this letter, we describe the fabrication, analytical techniques, and exemplary structures of the smallest (< 50 nm) compound semiconductor heterostructures. To accomplish patterning on such a fine scale, a variety of problems such as mask erosion, undercutting, resolution, and etch rate control had to be overcome. Some of the techniques used for the first time in III-V semiconductors include electron beam lithography at energies greater than 150 keV, fluoride etch masks and resists, and high-resolution reflection electron imaging of structures. Examples of small structures include arrays of < 40 -nm-diam "quantum disks" etched in GaAs/AlGaAs multiple quantum well material. Since the lateral dimension of these structures is close to the thickness of the quantum wells, photoluminescence spectra can be observed, which are very different from the grown layered material.⁵

We have established processes specific to the fabrication of ultrasmall, high-quality compound semiconductor samples. Electron beam lithography was used to define the small lateral dimensions.⁶ Electron beam voltages in excess of 150 keV were used to avoid broadening of individual disks through exposure to backscattered electrons and to minimize proximity effects or exposure variation over the sample. A scanning transmission electron microscope (STEM) was used to pattern the resist to attain beam sizes from 2 to 6 nm. Beam currents of 2–10 pA were obtained with a LaB_6 electron source, and the sample was focused in the previously aligned STEM by bringing the sample into the eucentric height, imaging a gold grid pattern under the polymethylmethacrylate (PMMA) resist with the backscattered electron detector.

Although negative resists can be used to directly expose

and, subsequently, etch the substrate, the most reproducible technique was a lift-off procedure using 70-nm-thick PMMA as a positive resist material. This resist was exposed by doses of $0.20 \text{ pC}/\mu\text{m}$. The resist was developed by a 10-s immersion in a 3:7 cellusolve:methanol mixture followed by a water rinse. A liftoff (Fig. 1) was used to provide the etch mask on the sample. To minimize the thickness of the etch mask and thereby gain more resolution in this lift-off procedure, either a 15-nm nickel or a 10-nm SrF_2 mask was used.

To structure the multiple quantum well material, reactive ion etching (RIE) was used. Since AlGaAs etches differently from GaAs, etching conditions which limit the lateral erosion of GaAs have to be chosen. This was accomplished in a 9:1 Ar: BCl_3 gas mixture at pressures of 15 mTorr at 50 W for a 6-in. substrate in a standard planar RIE system. These etching conditions were found to provide similar etch rates for AlGaAs and GaAs, and did not substantially undercut the GaAs. Etch rates of approximately 30 nm/min were obtained with these conditions.⁷

Scanning electron microscopy (SEM) and reflection electron microscopy (REM) were performed in a STEM

Liftoff Procedure

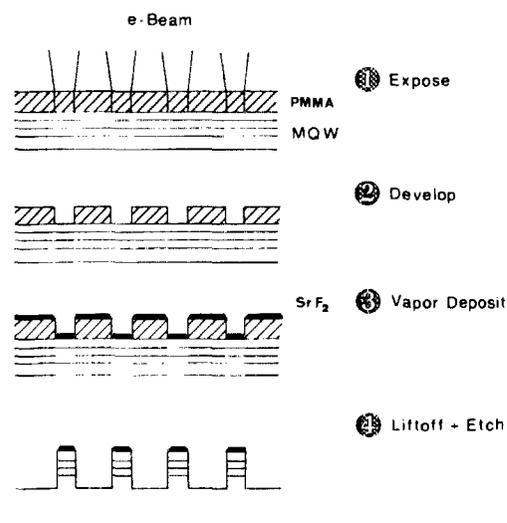
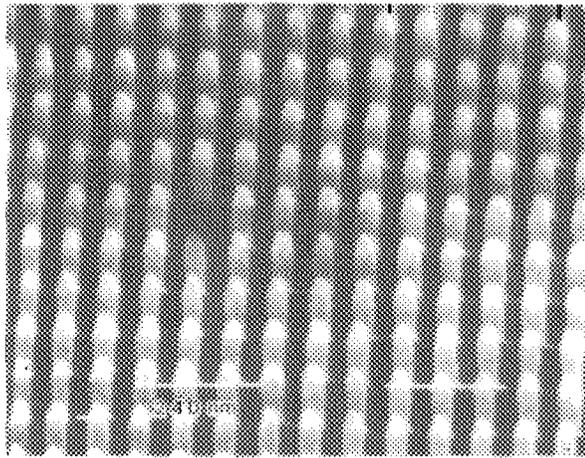
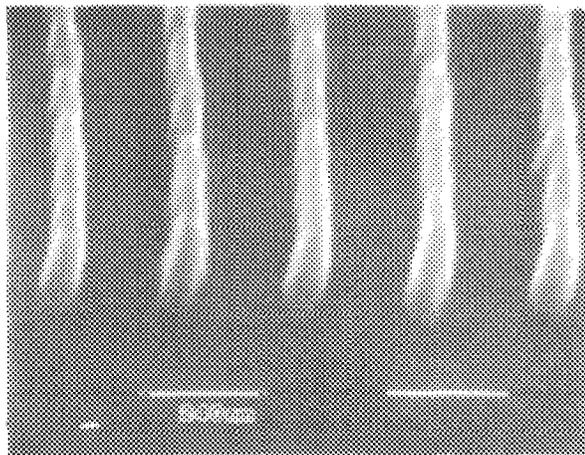


FIG. 1. Schematic of lift-off process used with PMMA resist.



(a)



(b)

FIG. 2. (a) SEM micrograph of columns etched into GaAs/AlGaAs multiple quantum well material, using SrF_2 as an etch mask. (b) SEM micrograph of lines etched into the same material (micrograph taken at 45°).

and were used to measure the dimensions of the patterned structures. The quality of the etched substrate was investigated with reflection electron diffraction both of patterned and of unpatterned areas. Photoluminescence spectra taken from overexposed and patterned areas and compared with the spectra of the original material (to describe the quality of the quantum wells after electron-beam irradiation) show an apparent increase in the luminescence efficiency of the patterned material.⁵

A sample consisting of several 15-, 6-, and 3-nm-thick quantum wells was patterned using SrF_2 as an etch mask. This sample was then etched 200 nm by RIE producing an array of columns with diameters of approximately 40 nm, as are shown in Fig. 2(a). Lines with dimensions of 50 nm were also produced and are shown in Fig. 2(b). For the analysis of the morphologies of structures with lateral dimensions of less than 40 nm reflection electron microscopy (REM), because of the high contrast and resolution,⁶ was used. In this technique, a dark-field image of the surface is obtained by tilting the electron beam into a bulk specimen until the specimen surface is almost parallel to the electron beam. A REM

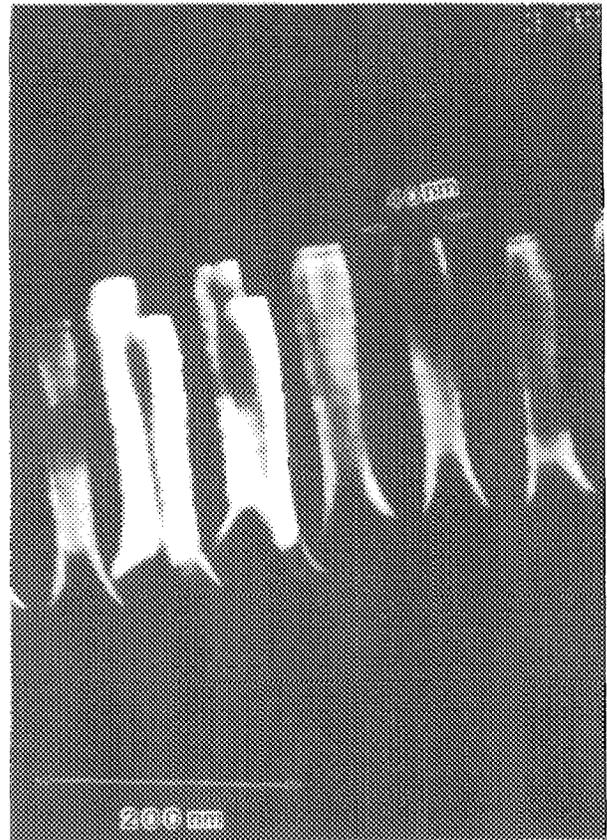


FIG. 3. REM image of columns etched into multiple quantum well material.

micrograph of etched columns is shown in Fig. 3. The dimensions of the structures can be determined accurately from this image using the etch depth, which is determined from depth profiles, as a standard. Lateral column dimensions ranging from 30 to 40 nm were measured in this way.

Contrast from individual quantum wells is obtained in the REM image, and a small amount of undercutting of the $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ layers can be seen in the form of steps on the sidewalls. This observation agrees well with reactive ion etch rate predictions for this gas mixture, according to which $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ etches slightly faster than GaAs under low power and BCl_3 concentrations used.⁷ However, contrast from the GaAs/ $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ superlattice is clearly visible throughout the column, indicating little disturbance of the sidewalls by the reactive ion etch. Reflection electron diffraction patterns, taken in the TEM, show bright Kikuchi line patterns on the etched surface. This is a further indication that only little damage of the material occurs in the top 10 nm of the sample.

Multiple quantum well structures have been patterned by electron beam lithography techniques to lateral dimensions of less than 50 nm, the smallest to date. A liftoff and reactive ion etching technique has been developed to reproducibly make such structures in GaAs/ $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ layers. This is accomplished by using novel etch masks and etch gas mixtures at conditions providing only minor differences in the GaAs and AlGaAs etch rates.

The measurement of the dimensions of the produced

structures, although still possible with standard SEM imaging, can be accomplished accurately by reflection electron microscopy. This technique also provides an understanding of the quality of the quantum wells, and is, therefore, very useful for analysis of the multiple quantum well structures.

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