Influence of chlorine on etched sidewalls in chemically assisted ion beam etching with SU-8 as mask

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Abstract. A thin layer of SU-8 submicron pattern produced by holographic lithography is used as the dry etch mask in a chemically assisted ion beam etching (CAIBE) system. The effect of the chlorine gas flow rate on etched sidewalls is investigated; by matching the lateral etch rate and the deposition rate, etching selectivity of up to 7:1 is achieved, rendering smooth vertical sidewalls and damage-free upper portions for the etched structure.
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Subject terms: dry etching; sidewall; SU-8; holographic lithography; duty ratio.

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1 Introduction

For optoelectronic device fabrication, it is important to control the pattern profile in the dry etching process accurately. In many cases, particularly in the subwavelength region, the etched sidewalls need to be vertical and smooth, while minimizing the damage during the etch process. Generally, this quality of profile can be achieved by carefully adjusting the parameters in reactive ion beam etching (RIBE),[1] where different arrangements have been made to produce the same profiles, namely, large ion beam divergence angle (about 24 deg) by Zubrzycki et al.,[1] and small ion beam divergence angle (about 1.5 deg) by Lincoln et al.[2] In the RIBE of GaAs with chlorine (Cl_2), ion beam sputtering removes the surface residues of $GaCl_x$ and $AsCl_x$ created during the process.[3] To achieve quality anisotropic etching with vertical and smooth sidewalls, the lateral etch rate needs to match the lateral deposition rate of the created residue. Meanwhile, since the upper portion of etched structure is easily damaged by the ion beam sputtering, a hard etch mask[1,4] or thick resist layers[5] were required to decrease the erosion of the mask layer and thereby protect the upper edge of the etched structure.

In this paper, we report the use of a thin layer of SU-8 photoresist patterned with nanostructures as a dry etching mask and the application of chemically assisted ion beam etching (CAIBE) to etch a GaAs substrate, emphasizing the fact that the key to achieving vertical and smooth etch profiles is to match the lateral etch and deposition rate by controlling the gas flow rate of chlorine.

2 Holographic Lithography

In our experiment, a holographic lithography system was used to pattern a nanoscale grating structure.[6] In our setup, an expanded and collimated Ar^+ laser beam (operating at $\lambda = 364$ nm) is split into two beams by a nonpolarizing UV beamsplitter. The two beams are then deflected onto the image plane at an angle θ, creating an interference pattern with a period of $\lambda/[2\sin(\theta/2)]$, which is adjustable by changing θ.

Before the exposure, SU-8 resist (MicroChem Corp.) is spin-coated on cleaned GaAs substrates, followed by a soft bake at a temperature of 95°C for 10 min to remove all the solvent in the SU-8 layer. After the exposure in the image plane of the holographic lithography setup, a postexposure bake (PEB) is applied to the sample to perform cross-linkage. The SU-8 is then developed in propylene glycol methyl ether acetate, rinsed in isopropyl alcohol, and dried in air.

3 Etching Experiment

The developed SU-8 structure is mounted on a wafer chuck in the vacuum chamber by a load lock system in our CAIBE setup, which is equipped with a 12-cm inductively coupled plasma (ICP) rf source with two self-aligned collimated graphite grids for screening and acceleration. The wafer chuck is located 45 cm away from the grids. The maximum beam and accelerator voltages are 2000 and 500 V, respectively; and typical values of the beam voltage range from 300 to 750 V, and of the accelerator voltage from 25 to 500 V, depending on the desired etch profiles. The base pressure of the vacuum chamber is pumped down to 10^{-8} Torr, and the typical chamber pressure during an etching process ranges from about 0.10 to 0.80 mTorr. The sample stage is temperature-controlled by a water chiller during the etching.

In our CAIBE, we use chlorine as the chemical etching gas and argon as the plasma ion (Ar^+) source. The etching rate and profile are influenced by many parameters in the system, such as the beam voltage, accelerator voltage, beam current, substrate temperature, chamber pressure, Cl_2 flow rate, and substrate material.[4,7] With a beam voltage of 400 V, accelerator voltage of 425 V, Cl_2 flow of 10 sccm (standard cubic centimeters per minute), Ar flow of 10 sccm, beam current of 40 mA, and substrate temperature of 5°C, a vertical sidewall profile was achieved for a 1-μm-

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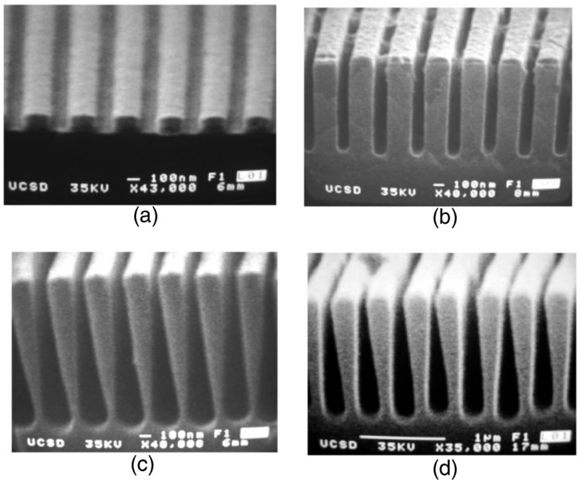

Fig. 1 SEM micrographs of 400-nm-period grating transferred to a GaAs substrate using CAIBE: (a) SU-8 resist mask with 150-nm thickness; (b) structure of 980-nm depth etched using 20-sccm Cl_2 flow; (c) 1480-nm depth using 18 sccm Cl_2 flow; (d) 1440-nm depth using 14-sccm Cl_2 flow.

period grating structure,[6] and the etching rate selectivity is approximately 3:1. With the same etch parameters, we have successfully transferred photonic crystals with hexagonal periodic patterns and defect lines to GaAs substrates.[8]

As the feature size of the devices scales down to submicron, surface tension becomes prominent when rinsing the sample during the development, reducing the achievable aspect ratio of the photoresist pattern, which is incapable of producing the etch depth for designed depth with the dry etch parameters described previously. Although one can use a rinsing solution with less surface tension and apply a supercritical drying procedure[9] to alleviate this problem, or transfer a patterned thin layer to a dry-etch-resistant mask layer[1,4] to improve the mask durability, both methods will increase the complexity and the cost of the process, and the patterns may degrade during the multiple processing steps involved in the latter.

Instead of dealing with the etch mask material, we optimize the dry etch parameters to perform high etch selectivity for a thin layer of submicron photoresist pattern. Based on the preceding conditions obtained for 1-μm pattern etching, we increased the stage temperature to 20°C, and reduced the Ar flow rate to 7 sccm to decrease the Ar^+ ion density. The Cl_2 flow rate was then changed to investigate its effect on the etched sidewall profile while the beam voltage (400 V), acceleration voltage (425 V), and beam current (40 mA) were kept constant.

We began with a Cl_2 flow rate of 20 sccm, corresponding to a chamber pressure of 0.68 mTorr. Figure 1(b) shows a scanning electron microscopy (SEM) image of the etched 400-nm-period GaAs grating with SU-8 pattern [as shown in Fig. 1(a)] as the dry etch mask. The thickness of the resist mask is about 150 nm, with duty ratio (ratio of the grating tooth width to the period) of about 60%, and the etched depth into the GaAs is about 980 nm. The aspect ratio of the etched pattern is about 4:1, with an average etching rate of 20.5 nm/min. As we can see in Fig. 1(b), the etched sidewalls are almost vertical; however, there are a lot of residues in the grating grooves, which would strongly affect the optical characteristic of the device. The residues are the redeposited reactants of $GaCl_x$ and $AsCl_x$ that cannot be removed with Ar^+ ion bombardment in an acceptable time. The residue formation is pronounced with excessive chlorine, which raises the local pressure and increases the redeposition rate within the grooves.

In order to avoid the contamination, the Cl_2 flow rate was decreased to 18 sccm, corresponding to a chamber pressure of 0.60 mTorr, while the other parameters were kept unchanged. As shown in Fig. 1(c), the etching depth is 1480 nm. The etched profile, however, is inversely tapered instead of rectangular, with a duty ratio decreasing from 64% in the upper portion to 19% in the bottom portion. The smooth inversely tapered shape indicates that the lateral etch rate is greater than the redeposition rate of the reactants. Unlike the case in Fig. 1(b), there are no residues between the grating grooves, showing that the etching reactants are removed and pumped out from the sample surface efficiently.

In order to approach rectangular etched profiles, the lateral etching rate can be reduced by decreasing the flow rate of Cl_2. Figure 1(d) shows a sample that is etched using a reduced Cl_2 flow rate of 14 sccm, with a corresponding chamber pressure of 0.45 mTorr; the etch depth is about 1440 nm. Although the profile is still inversely tapered in this particular case, the duty ratio of the bottom portion increases to 30%, with the duty ratio of the upper portion unchanged from the profiles in Fig. 1(c). This shows that we have a good balance between the lateral deposition rate and the etching rate in the upper portion of the etched structure with the applied etch parameters, and changing the Cl_2 flow rate exhibits little effect on this portion.

On further reducing the lateral etch rate by decreasing the Cl_2 flow rate, a rectangular etch profile can be obtained. Figure 2(a) shows an etched structure using a Cl_2 flow rate of 7 sccm with a 0.22-mTorr chamber pressure. In this particular case the SU-8 mask layer is a little too thin for the long process duration (more than 100 min); therefore the resist had been etched out and the top surface was slightly overetched. However, it is evident that the profile (although still slightly inversely tapered) improves significantly, with the duty ratio of the etched bottom portion increased to 45%.

After optimizing the Cl_2 flow rate (at 6 sccm with the chamber pressure of 0.18 mTorr), we obtained a sample with rectangular etch profiles as shown in Fig. 2(b). The etched sidewalls are vertical and smooth, with a depth of about 790 nm and a duty ratio of 46% (same as the upper portion). As one can see in Fig. 2(b), for this particular sample, although the resist remaining on the top of the grating structure is no longer rectangular, the top portion of the etched pattern is kept rectangular, which is another advantage of this optimal dry etching process.

To summarize the effect of the Cl_2 flow rate on the etch profiles, an *undercut angle* is defined as the angle between the etched sidewall and the normal to the substrate surface. On reducing the flow of Cl_2 from 18 to 6 sccm, or the chamber pressure from 0.60 to 0.18 mTorr, the undercut angles of the etched profile decreased from 7 to 0.2 deg, as shown in the plot of Fig. 3(a), and the profile of the etched

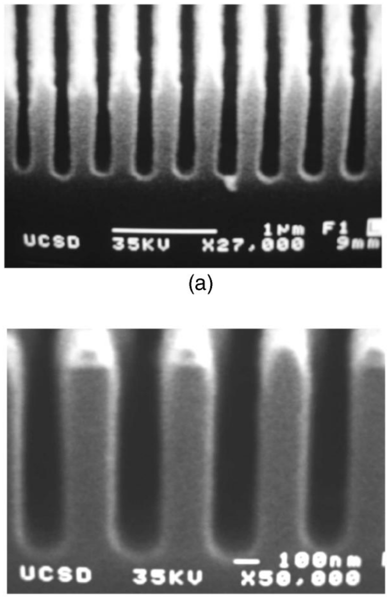

Fig. 2 SEM micrographs of GaAs grating structure with period of 400 nm etched in CAIBE using SU-8 resist layer as mask: (a) overetched structure using 7-sccm Cl_2 flow; (c) smooth vertical etched structure with 790-nm depth using 6-sccm Cl_2 flow.

structure changed from inversely tapered into almost rectangular. It is obvious that on further reducing the Cl_2 flow rate, the undercut angle of the etched profile would even decrease to negative values, that is, the etched profile would become tapered, and indeed, that is how most etched structures of tapered shape resulted. However, when the Cl_2 flow rate was increased to 20 sccm (pressure of 0.68 mTorr), instead of a worse tapered shape, the undercut angle of the etched profile suddenly dropped to 1.8 deg with almost vertical but rough sidewalls. The redeposition of the reactants is now playing an important role in keeping the sidewalls vertical; reactants could not be removed effectively by bombarding Ar^+ ions and thus were redeposited on the sidewall, preventing the profile from becoming worse tapered. If the residues in the grating grooves can be removed afterwards, this procedure is also possible approach to producing the desired profiles.[10]

As the Cl_2 flow rates change, however, the average etching rate, defined as the ratio of etched depth to etching duration, remains almost unchanged at about 20 nm/min, as shown in Fig. 3(b), indicating that the etching rate is mainly attributable to ion beam sputtering in our low-intensity ion beam etching, but not in the cases of high-density plasma[7] and high ion energy,[2] in which the etching rate strongly depends on the Cl_2 flow rate. For our optimal etching process, the erosion rate of the SU-8 resist and the etching rate of GaAs are about 3 and 20 nm/min, respectively, so that the etching-rate selectivity is approximately 7:1. With a 150-nm-thick SU-8 mask layer, about 1-μm depth into GaAs can be achieved, which would be enough for our polarization control devices in communication applications.[10] For deeper etching, a thicker SU-8 layer can be achieved by use of supercritical resist-drying methods,[9] which we are currently investigating.

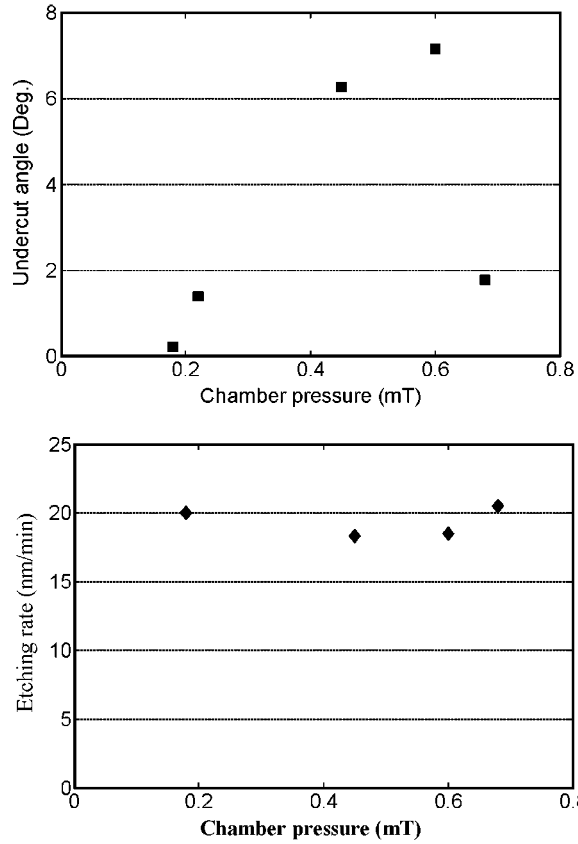

Fig. 3 Dependence of (a) the undercut angle of the etched profile and (b) the etching rate of GaAs on the chamber pressures.

4 Discussion and Conclusions

Although the sidewalls of the etched grating structure strongly depend on the Cl_2 flow rate, the etching characteristics of transferring 2-D gratings with hole structures are somewhat different. In the transfer of a hole structure from SU-8 resist into GaAs, the shape of the etched sidewall is less sensitive to the Cl_2 flow rate than in 1-D grating etching, maybe because less area needs to be etched; the physics behind this will be investigated in future work. Furthermore, due to the stability of the web network of the mesh structure, we can achieve a very thick mask layer without being worried about the collapse as in the case of the 1-D resist grating; other etching parameters, such as the beam voltage and beam current, also could be changed to get faster etching rates.[8]

The duty ratios of SU-8 mask layers, which we are using to test the etching process, fluctuate between 46% and 64% due to the different exposures.[6] If the duty ratios vary too much among different samples, or for very small feature size, the size effect may need to be considered.

In conclusion, we have used a thin layer of SU-8 photoresist pattern produced by holographic lithography as a dry etch mask to etch GaAs substrates in a CAIBE system. By optimizing the flow rate of chlorine to match the lateral etch rate and deposition rate, etching selectivity of up to 7:1 has been achieved with smooth vertical sidewalls and damage-free upper portions of the etched structures. As a fast, easy, and low-cost method, this processing holds promise for fabrication of large-area submicron structures into Ga(Al)As materials, which, combined with optical lithography, are being used to fabricate large-area polarization-sensitive lenses for communication wavelengths.

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