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PHOTOELECTROCHEMICAL ETCHING OF BLAZED ESCHELLE GRATINGS IN n-GaAs

by

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Photoelectrochemical etching can be a technique for producing microstructures in semiconductors with a high aspect ratio and excellent lateral uniformity. We had demonstrated previously that symmetrical V-groove Eschelle diffraction gratings, used in a variety of spectrometers and opto-electronic devices, can be fabricated by photoanodic dissolution of (100) oriented GaAs, using a Ronchi ruling photoresist mask. In this paper, we report the etching of blazed Eschelle gratings of 15 x 15 mm dimensions and with 50 cycles/mm. To do this, n-GaAs crystals were sliced with a (100)-n° orientation, with respect to the (011) plane. By varying the angle n, gratings with blaze angles of 45, 53 and 60° have been demonstrated. *In situ* coulometry was used to monitor the etching process and to determine when the grating reached completion. SEM and optical measurements of the blaze angles of the completed gratings were in close agreement.

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PHOTOELECTROCHEMICAL ETCHING OF BLAZED ESCHELLE GRATINGS IN N-GaAs

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INTRODUCTION

Photoelectrochemical etching can be a technique for producing microstructures in semiconductors with a high aspect ratio and lateral uniformity. As an example of this application, we have reported recently on producing sawtooth gratings in (100) oriented crystals of n-GaAs using this method [1]. The process takes advantage of the orientational dependence of photoelectrochemical dissolution of GaAs, which favors the (111)As over the (111)Ga polar face, similar to some types of oxidative chemical etching [2,3]. Etching in the (100) surface necessarily results in symmetrical groove profiles, while most optical applications of gratings require unsymmetrical blazed structures.

It is the purpose of this paper to demonstrate photoelectrochemical etching of blazed, deep Eschelle gratings. These gratings are employed in a variety of electro-optical devices and in very high resolution spectrometers. Because of the large amount of material which must be removed in the fabrication, they are difficult to produce with smooth walls by conventional ruling engines, particularly in the low groove densities frequently needed.

EXPERIMENTAL

GaAs wafers (n-type, 5×10^{17} cm^{-3}) were cut from a boule supplied by Bertram Laboratories. Diced wafers of 5/8 x 5/8" dimensions were mounted as electrodes and then sequentially polished with alumina abrasives down to 0.05 µm, and finally chemomechanically polished with a silica/bleach slurry. Grating patterns were produced in positive photoresist (Shipley 1350J) with a periodicity of 50 cycles/mm. A line to space ratio of 2 was determined to give optimum results [4]. Both the photoresist exposure and the photoelectrochemical etching were accomplished using a highly collimated UV source (Oriel Corporation 87301 Illuminator). All experiments were conducted with potentiostatic control in a 3-electrode cell with a standard calomel (SCE) reference electrode.

RESULTS AND DISCUSSION

In order to produce blazed Eschelle gratings, it is necessary to cut the GaAs crystal at an angle off the (100) plane toward the (011) plane. As shown in Figure 1, orienting the photoresist lines in the $[01\bar{1}]$ direction should then give rise to structures with the interior angles governed by the preferred Ga-rich surfaces. One advantage of photoelectrochemical etching for producing these structures is that the process can be followed coulometrically. The charge, Q, required to etch the V-groove sawtooth pattern is:

$$Q(C/cm^2) = 3.54 \times 10^3 \, nN \left(\frac{0.5\,W^2}{\cot(\alpha-\beta)+\cot(\alpha+\beta)} \right) \quad (1)$$

Here, n is the electron stoichiometry (equivalents/mole) of the photoanodic dissolution reaction, W is the width (cm) of each groove, α is the angle of the groove face with respect to the (100) surface, β is the angle of the crystal slice with respect to the (100) surface, and N is the number of grooves/cm. In the present case, n = 6, N = 500 and $W = 2 \times 10^{-3}$ cm.

To demonstrate the photoelectrochemical etching of blazed structures, crystals were cut with (100), (100)-8° and (100)-18° orientations. The electrolyte composition was 0.1M KCl, adjusted to pH 3, and the light intensity was 30 mW/cm^2. The potential was held at the onset of the photon limited region, 0.4V vs. SCE. Initial structures were etched under the assumption that the interior angles were 70.54°, as defined by the (111) Ga surfaces. However, we found under closer examination that this angle was dependent on the etching conditions and on the electrolyte, and was closer to 90° under the present conditions. This would correspond most closely to the (223)Ga-rich surface. With a 90° interior angle, equation (1) predicts a charge of 10.6 and 8.6 C/cm^2 required to etch the gratings in the (100) and (100)-18° degree surfaces, respectively. With coulometric monitoring, both unblazed and blazed gratings were produced with pointed tops and bottoms and extremely smooth walls. A scanning electron micrograph (SEM) of the blazed structure from the (100)-18° surface is shown in Figure 2. The blaze angle of 60° is slightly less than the expected value of 63°, an error probably due to inaccuracies accumulated in the cutting and polishing procedures.

Since making cross sections for SEM analysis is a destructive technique, an optical method was developed for routine determination of the blaze angle. The gratings were mounted on a graduated turntable with the grooves parallel to the rotation axis, and illuminated with a He-Cd laser source (442 nm). The zero order reflection was used as a reference point, i.e., when the grating is 90° with respect to the laser source. The grating was then rotated and the angles recorded which produced a back-reflected beam that passed the laser aperture. The angle of the brightest back-reflection is the blaze angle (θ_1). It can also be calculated from the order number (m) giving this strongest back reflection, according to the grating equation

$$m\lambda = 2d\sin\theta \qquad (2)$$

where d is the groove spacing and λ the wavelength. Table 1 summarizes the blaze angles for several gratings measured from the turntable angle, from equation (2) and from SEM cross section profiles. Also included in Table 1 are the complementary blaze angles, θ_2, measured by rotating the turntable in the opposite direction ($\theta_1 + \theta_2 \sim 90°$). It is seen that the three methods give results that are in excellent agreement.

ACKNOWLEDGMENT

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Table 1. Properties of Photoelectrochemically Etched Eschelle Gratings

Sample	Orders for θ_1/θ_2	Blaze Angle θ_1/θ_2 Turn-table	Eq.2	SEM	Theory
1	63/64	43/45	44/45	43/44	45/45
2	64/65	44/46	45/46	44/45	45/45
3	71/56	53/37	52/38	51/39	53/37
4	70/57	52/39	51/39	51/39	53/37
5	78/48	59/31	60/32	60/30	63/27

Sample key:
1. (100), 0.5 M KCl
2. (100), 0.1 M KCl
3. (100)-8°, 0.1 M KCl
4. (100)-8°, 1.0 M KCl
5. (100)-18°, 0.05 M KCl

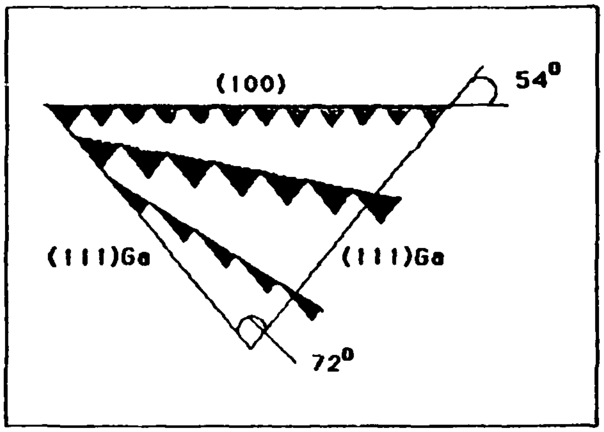

Fig. 1. Cross sections of V-groove formed by photoelectrochemical etching of (100)n-GaAs slots defined in the [01Ī] direction, and in two surfaces cut at intermediate angles between the (100) and (111)Ga surfaces.

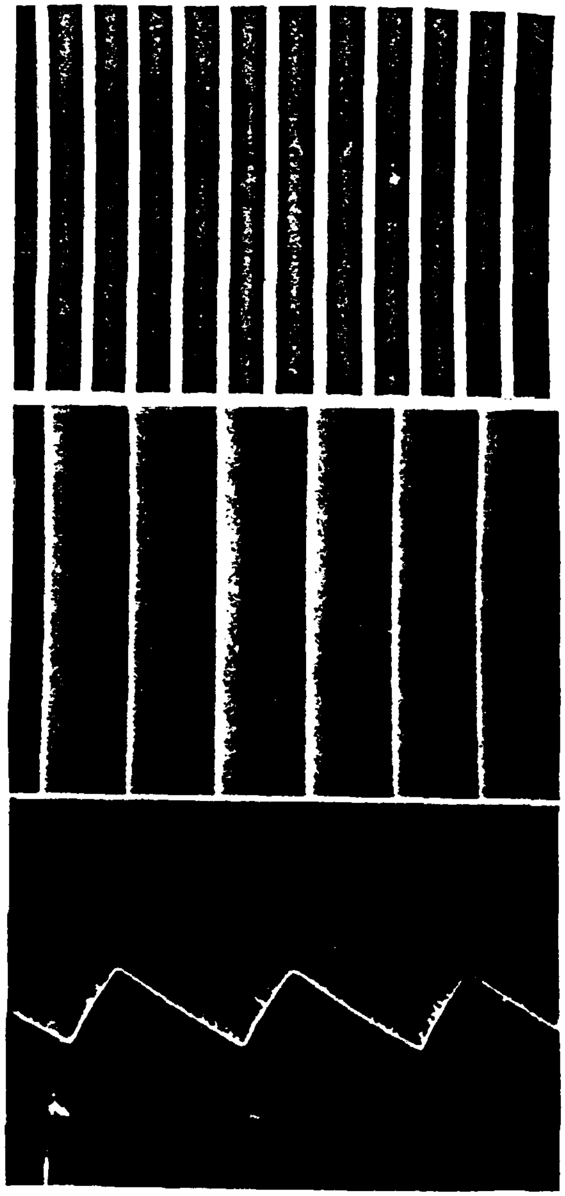

Fig. 2. Scanning electron micrographs of blazed Eschelle gratings etched photoelectrochemically in the (100)-18° surface of n-GaAs. Groove spacing is 20 µm.

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