Infrared Grisms Using Anisotropic Etching of Silicon to Produce a Highly Asymmetric Groove Profile

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ABSTRACT

Grisms are an important tool for astronomical spectroscopy because they allow for very compact, straight-through spectrometer designs in systems that can double as imagers. In the infrared, silicon grisms offer the advantage of superior resolving power for a given beam size and opening angle, when compared to grisms made of low refractive index materials. Silicon grisms with symmetric profiles and a blaze angle of 54.7°, the natural result of anistropic etching of silicon substrates oriented with the (100) crystal plane exposed, are relatively easy to produce. Low-order grisms, however, must be blazed at much shallower angles and will therefore have highly asymmetric groove profiles. In order to achieve these shallow blaze angles, the silicon surface must be precisely oriented at a bias from the (100) plane before cutting and polishing the substrate. Production of gratings with blaze angles as small as 6° is more difficult than production of unbiased gratings because it is very sensitive to changes in the etching process parameters. In this paper, we discuss our techniques for etching highly biased surfaces in silicon wafers, along with the first results of our production and testing of highly biased silicon gratings, including SEM groove profile pictures and optical testing in reflection at 632 nm and in transmission at 1523 nm.

Keywords: silicon grism, anisotropic etching, asymmetric groove profile

1. INTRODUCTION

Grisms, transmission gratings cut into the surface of prism-shaped dielectric substrates, are an important element in the designer's toolbox for astronomical spectrometers. When grisms are blazed at an angle matched to the opening angle of the prism, the wavelength at the center of each order continues in the direction of the incident collimated light. If both the opening angle of the prism and the incidence angle are small, the performance of the grism is insensitive to its orientation in the beam, thereby easing alignment problems. These features of grisms make it possible to build simple systems that serve as both imagers and spectrometers. The diffraction-limited resolving power of a grism with a given size and opening angle scales with the refractive index of the dielectric material as n-1. This scaling means that a grism made from a high index material like silicon (n=3.44) can have a resolving power almost five times higher than that of a grism made from a low-index glass.

Optical and short wavelength near-IR grisms are usually produced by replicating a ruled grating into a soft resin adhered to one side of a prism. This technique does not work well at longer wavelengths because of the high IR absorptivity of the resin material and the poor match of its refractive index to the high index materials which are so desirable in the mid-IR.[1] For near-IR work, it has also been possible to rule gratings directly onto KRS-5 prisms.[2,3] Efforts at direct ruling of other materials have been less successful.

The difficulty of ruling many of the best materials for infrared grisms lead researchers to investigate the possibility of making grisms on silicon substrates using micromachining techniques. The very earliest diffraction gratings made by anisotropic etching of silicon were etched across the (100) plane and had symmetric groove profiles.[4] Over the past decade, work toward astronomically useful gratings produced by micromachining symmetric grooves into silicon has progressed significantly.[5–11] Our own recent gratings have excellent groove surface quality and efficiencies comparable to those of ruled gratings.[10,12,13]

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Grism spectrometers used for the initial spectroscopic followup to imaging observations often need broad spectral coverage at modest resolving power. The need for broad coverage means that the gratings must operate in low order. In the mid-IR, where the array sizes are still modest, however, the number of resolution elements per order cannot be too large if all of a given order is to fit on the array. This second requirement leads to a need for fairly coarse grooves which, since they are to work in low order, must be blazed at quite shallow angles.

We have calculated strawman designs for a number of potential near-IR and mid-IR grism spectrometers. One set of grisms for use in the FORCAST mid-IR camera on SOFIA would need to cover the spectral range 5 - 40 μm and permit spectroscopy at low resolving power (R$\sim$100 - 5000). The optimal design for a FORCAST grism with a diffraction-limited resolving power R=$\lambda/\Delta\lambda$=1000 at 5.5 - 8.5 μm would operate in first order and have a groove spacing of 25 μm. This grism would need to be blazed at 6.6°. To produce this device, we would need to begin with a substrate oriented so that a surface tilted by 48.1° from the (100) plane (and only 6.6° from the (111) plane) is exposed. For R=400 at 19 - 25 μm, we came up with a design having a groove spacing of 142 μm and a blaze angle of 11°.

The small blaze angles in our FORCAST designs require highly asymmetric groove profiles. A limited number of earlier papers describe the use of anisotropic etching techniques to produce gratings with the highly asymmetric groove profiles necessary to make grisms with small blaze angles.[14–17] These authors applied their techniques for the production of asymmetric grooves only to fine gratings covering small areas of Si wafers. They did, however, investigate some of the key issues involved in producing etched gratings with asymmetric grooves. Among the most important of these are the need to cut the Si substrates at the correct orientation to produce the desired blaze (inclined by a specified amount with respect to one of the crystal axes) and the need to orient the photolithography mask precisely with respect to the crystal structure of the Si substrate. Both of these requirements push existing x-ray diffractometry facilities to their limits.

If the particular problems involved in producing micromachined gratings with highly asymmetric profiles can be overcome, the prospects for producing high quality grisms for near-IR spectroscopy are quite good. The requirement on the positional accuracy of the grooves in low-blaze angle grisms, the most difficult specification to meet when making etched diffraction devices, is much less stringent than for immersion gratings. The accuracy requirement for groove position along the patterned surface is reduced compared to immersion gratings operating at the same wavelength by a factor of $f = [2n/(n-1)] \times [sin\delta/sin\gamma]$ where δ is the blaze angle of the immersion grating and γ is the opening angle of the grism. For a silicon grism with an opening angle of 10° compared to an R=2 (δ=63.2°) immersion grating, f=14.5. Given the groove positional accuracies we have achieved for silicon gratings etched into both wafers and bulk Si substrates,[10, 12] it should be possible to use micromachining techniques to produce good quality Si grisms throughout the range in the infrared where silicon is transparent.

In this paper, we describe the production of coarse gratings with highly asymmetric groove profiles on Si wafers, show the resulting pieces, and use optical and IR spectroscopic techniques to evaluate the performance of the completed devices.

2. FABRICATION

The basic steps in production of gratings with asymmetric groves are similar to those for gratings in which the grooves are symmetric. Figure 4 of Jaffe et al. (1998) illustrates the micromachining processing steps we use to fabricate symmetric grove gratings. We began our fabrication with two batches of silicon wafers sliced at 6.6° and 10° angles from the (111) plane of a float zone grown silicon ingot, exposing surfaces tilted by 61.3° and 64.7° from the (100) plane. The 0.5 mm thick wafers were obtained from Silicon Quest International. Each wafer was provided with a reference flat at the intersection of the (100) and (111) crystal planes. The wafers were coated with either 600 nm silicon dioxide or 100 nm silicon nitride. For wafers with an exposed (100) surface, we have normally made a precise definition of the line of intersection of the (100) and (111) crystal planes by scoring the surface along a line parallel to a roughly accurate orientation flat milled into the surface by the manufacturer and then snapping the wafer along this line.[12] The extreme bias of the wafers used in the present study made it impossible to score and cleave them cleanly along the intersection of the (100) and (111) planes. For these wafers, it was necessary for the substrate manufacturer to measure a precise orientation using x-ray crystallography, mill an orientation flat and use this orientation flat to align the mask. The specifications for the wafers required that the orientation flat be within ±0.05° of the intersection between (100) and (111). We were not equipped, however, to verify the accuracy of the alignment flats independently. In the photolithography process, the contact mask was aligned optically with the orientation flat before exposing the photoresist.

In crystalline silicon, the intersection of (111) planes forms angles of 109.5° and 70.5°. In both cases, the deviation of the vertices from a right angle results in shadowing of a given groove by its neighbor. For small blaze angles, this is a minor effect, but it becomes quite significant for blaze angles >30°. For grisms, light lost to blockage by adjacent grooves will be lower if the exposed surface lies within one of the 70.5° vertices (see Figure 1). In grooves with 109.5° vertices, the width of the photolithographic etch stop stripes add to the vertex shadowing while it does not do so for the grooves with 70.5° vertices (exposed surface 1). The wafers used to produce the devices we discuss here, had exposed surfaces which lay within one of the 109.5° vertices (exposed surface 2). This orientation results in groove profiles with 109.5° opening angles (Figure 2).

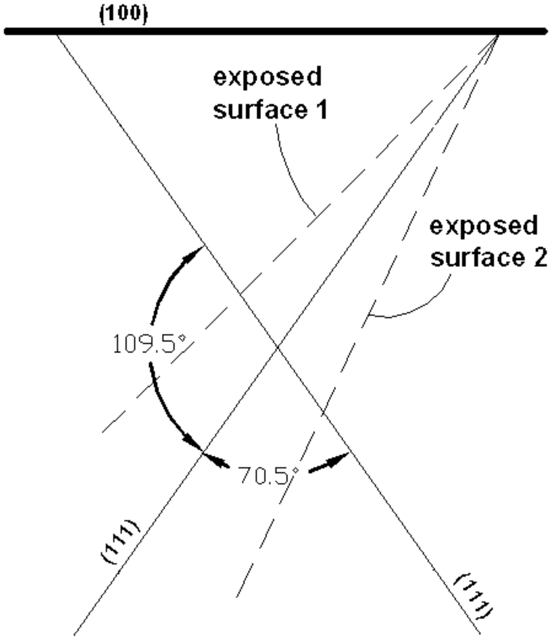

Figure 1. Two possible orientations for the exposed surface in a silicon wafer cut at a bias of 10° from the (111) crystal plane. Surface 1 lies 44.7° from the (100) plane while surface 2 is 64.7° from this plane.

We experimented with patterning the wafers using both a silicon dioxide passivation layer with buffered oxide etching and a silicon nitride passivation layer with reactive ion etching (RIE). The patterning of silicon dioxide is described in our previous work.[10,12,18] The benefits of using RIE for pattern transfer are two-fold. RIE is highly anisotropic, thereby reducing angular error in the pattern transfer step.[18,19] It also patterns the hardier Si_3N_4, allowing the passivation layer to be thinner (100 nm instead of 600 nm for SiO_2). Among our wafers, Si_3N_4 layer thicknesses varied from 98 to 104 nm (determined using an ellipsometer). We etched away 110 nm, according to the Si_3N_4 RIE rate, to ensure proper removal of the Si_3N_4 layer while not overetching into the silicon.

After patterning, wafers passivated with both silicon dioxide and silicon nitride underwent anisotropic etching in an alkaline solution (KOH) using techniques described in our earlier work.[10,12,18] The etching times per unit depth for the highly asymmetric groove profiles, however, were considerably longer than for the etching of symmetric grooves described earlier. Using our established recipe, it takes 60 minutes to etch a 22 μm wide groove, starting from an exposed (100) crystal plane resulting in a symmeric V-shaped groove that is 18 μm deep (etch rate 0.3 μm per minute). If one uses the same 25 μm mask to pattern a wafer where the exposed surface lies 64.75° from the (100) plane and only 10° from the (111) plane, it takes 50 minutes of etching to produce the 4 μm deep asymmetric groove (etch rate 0.07 μm per minute).

When etching symmetric grooves into substrates with SiO_2 passivation, one expects the groove to undercut the original striped SiO_2 pattern (in a direction parallel to the (100) surface). SiO_2 is isotropically etched at a rate of ~0.4% of the Si (100) etch rate.[20] The edges of the passivation layer stripes are etched away, further exposing the (100) plane which produces uneven groove walls toward groove tops. Additional undercutting occurs due to the finite (albeit small) etching rate in the <111> direction. Ratios of (100) to (111) etch rates vary depending on the KOH concentration and temperature of the wet etching solution, but a typical (100) to (111) etch rate ratio is 200:1.[21] The undercutting was uniform enough, however, that it did not strongly affect the precise locations of the grooves on gratings with symmetric groove profiles. Gratings produced using Si_3N_4 passivation, for which no etching of the passivation layer occurs, were only marginally better.[12] The effects of undercutting are more severe for highly asymmetric groove profiles than for symmetric profiles. The increased undercutting could be due to a faster (111) etch rate depending on orientation.[22] Undercutting due to the etching away of the SiO_2 etch stops is much deeper than for the symmetric grooves. We found that using silicon nitride passivation layers generally led to less undercutting and better groove positional accuracy for these devices, particulary for gratings with wider groove spacing.

3. TESTING

For samples with 142 μm groove spacing, it is possible to take a picture through a microscope with a digital camera to verify groove profile geometry (Figure 2). Figure 2 shows the 109.5° vertex angle. In Figure 3, we compare SEM pictures of a device with an asymmetric groove profile with 25 μm spacing and a device with a symmetric groove profile produced from the same mask.

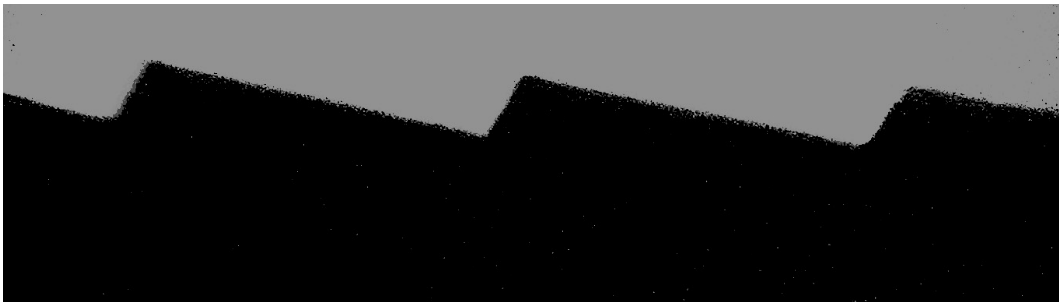

Figure 2. Photograph of groove ends for a grating with 142 μm grooves and an asymmetric profile for a 10° blaze. The opening angle at the groove bottom is 109.5° for this device.

Our original test setup used red and green HeNe lasers to test our gratings as front-surface devices. It consisted of a Twyman-Greene interferometer which could also be used as a spectrometer by adding a camera lens to focus the light onto the CCD camera.[12] We recently acquired a near infrared camera (Indigo Systems Alpha NIR model) thus supplementing our front-surface optical tests with near-IR transmission tests. The infrared testing scheme is described in detail in our other paper published in this volume.[23] At present, we did not use antireflection coatings for our transmission gratings.

We tested our biased gratings (6.6° biased with 25 μm groove period and 10° biased with 142 μm groove period) both at 632 nm in reflection and at 1523 nm in transmission. The spectra are shown in Figures 4-6. Throughput was measured in reflection using the same procedure described in our previous papers[10, 12, 18] for our grating with 142 μm groove spacing and 10° blaze angle. At 632 nm, the measured throughput in all observed orders was 72%. Approximately 12% of incident light was lost due to shadowing from groove tops and unused groove walls, so that the maximum achievable throughput was 88% for the given geometry. Because

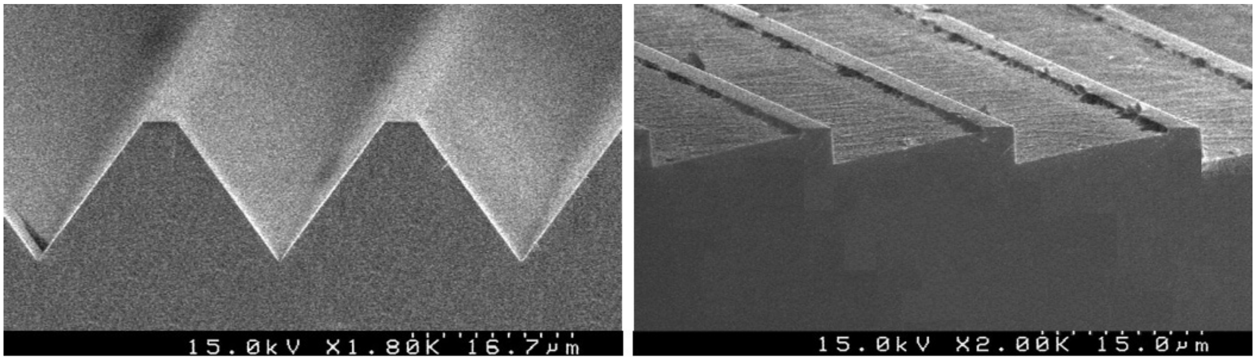

Figure 3. (left) SEM photograph of groove ends for a grating with 25 μm grooves with a symmetric profile. The flat stripes along the top of the image are the remainder of the etch-stop stripes patterned into the <100> crystal surface. The opening angle at the bottom of the grooves is 70.5°. (right) SEM photograph of a grating with 25 μm grooves and a highly asymmetric profile (designed to produce a blaze angle of 10°). In this case, the opening angle at the groove bottom is 109.5°.

our wafer grating is not optically flat, light is diffracted into several orders. The throughput in the peak order at the blaze wavelength would have been about 65% if the grating were etched on an optically flat substrate.

Diffuse scattered light results from imperfections in groove walls or, in the case of transmission gratings, from imperfections in crystal structure. The estimated amount of diffuse scattering in our biased gratings is 2-3% for 142 μm period grating and 3-4% for 25μm period grating. The low amount of scattering in our gratings indicates that the quality of the grooves is sufficient for astronomical applications.

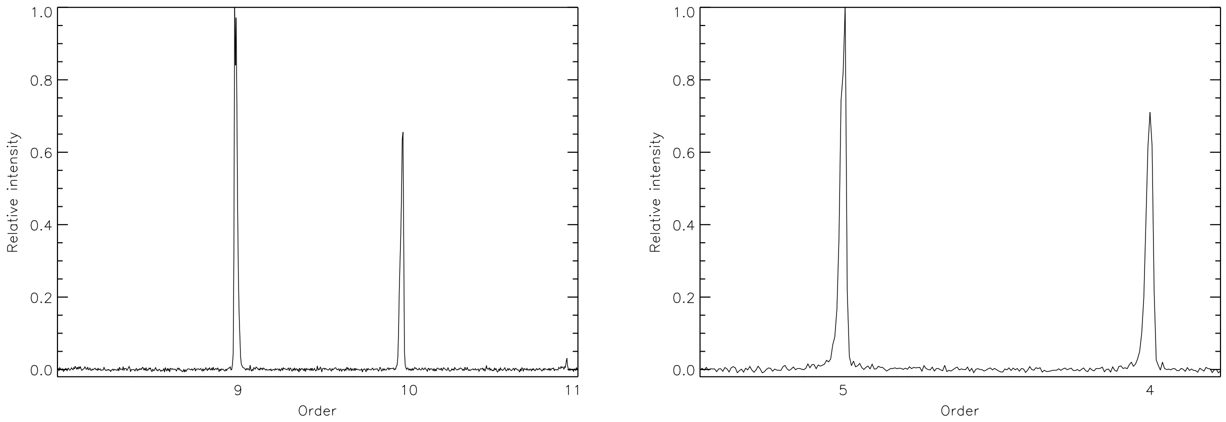

Figure 4. (left) Diffraction spectrum at 632 nm in reflection for a 25 μm echelle blazed at 6.6°. (right) Diffraction spectrum at 1523 nm in transmission for a 25 μm echelle blazed at 6.6°. The samples tested here were produced with a low quality e-beam lithography mask.

4. CONCLUSION

This work demonstrates that it is possible to make optically efficient gratings with highly asymmetric groove profiles using silicon micromachining techniques. This capability opens up the possibility of producing silicon grisms with any desired blaze angle.

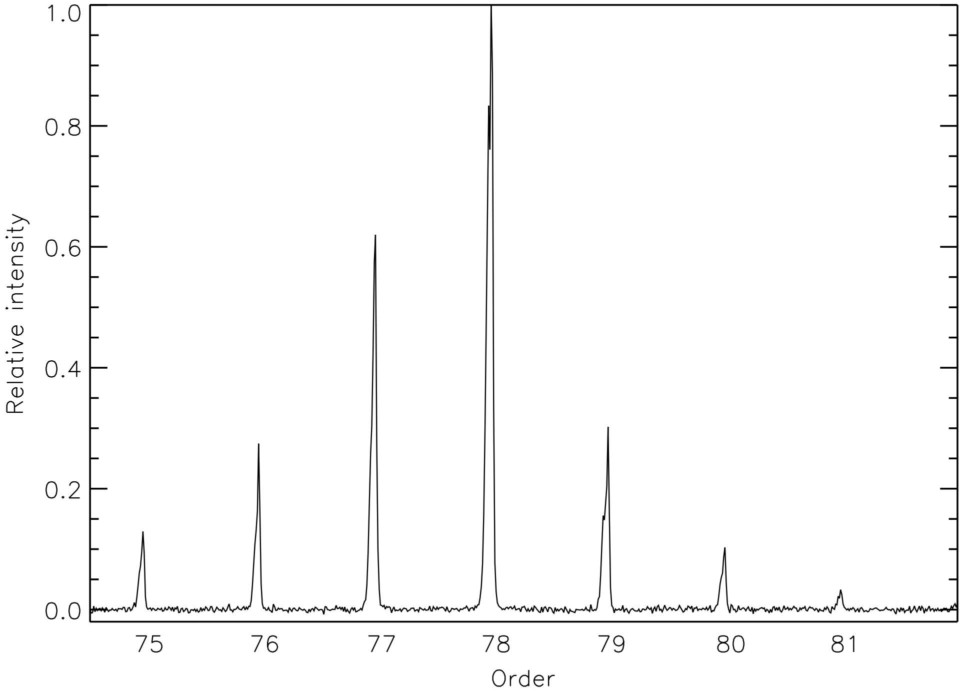

Figure 5. Diffraction spectrum at 632 nm (reflection mode) for 142 μm echelle blazed at 10°

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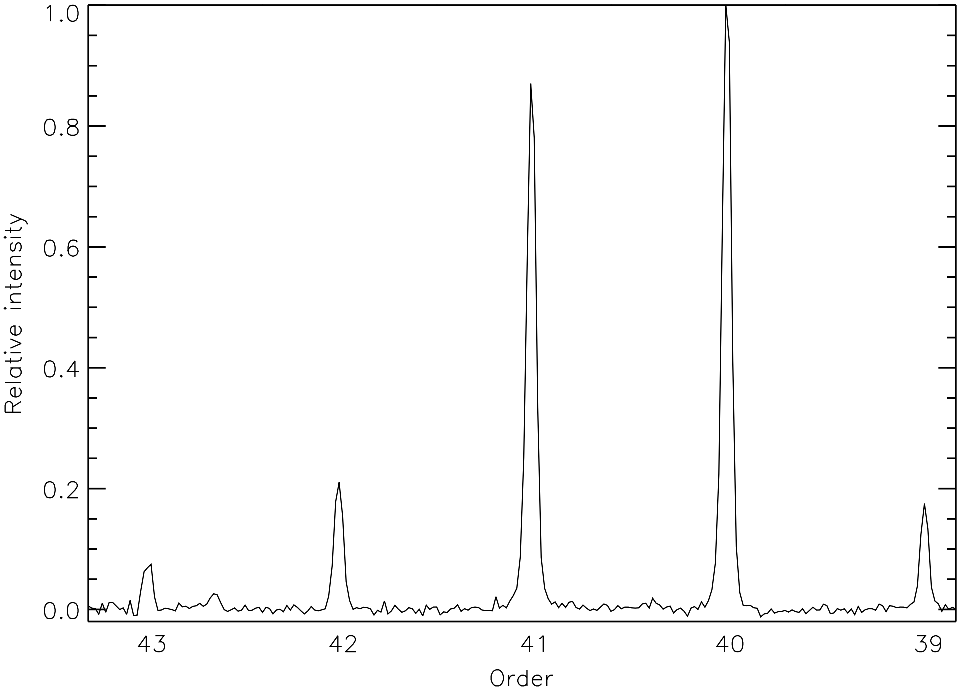

Figure 6. Diffraction spectrum at 1523 nm (transmission mode) for 142 μm echelle blazed at 10°

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