Development of Silicon Grisms and Immersion Gratings for High Resolution Infrared Spectroscopy

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ABSTRACT

We report new results on silicon grism and immersion grating development using photolithography and anisotropic chemical etching techniques, which include process recipe finding, prototype grism fabrication, lab performance evaluation and initial scientific observations. The very high refractive index of silicon (n = 3.4) enables much higher dispersion power for silicon-based gratings than conventional gratings, e.g. a silicon immersion grating can offer a factor of 3.4 times the dispersion of a conventional immersion grating. Good transmission in the infrared (IR) allows silicon-based gratings to operate in the broad IR wavelength regions (~1-10 µm and far-IR), which make them attractive for both ground and space-based spectroscopic observations. Coarser gratings can be fabricated with these new techniques rather than conventional techniques, allowing observations at very high dispersion orders for larger simultaneous wavelength coverage.

We have found new etching techniques for fabricating high quality silicon grisms with low wavefront distortion, low scattered light and high efficiency. Particularly, a new etching process using tetramethyl ammonium hydroxide (TMAH) is significantly simplifying the fabrication process on large, thick silicon substrates, while providing comparable grating quality to our traditional potassium hydroxide (KOH) process. This technique is being used for fabricating inch size silicon grisms for several IR instruments and is planned to be used for fabricating ~ 4 inch size silicon immersion gratings later.

We have obtained complete K band spectra of a total of 6 T Tauri and Ae/Be stars and their close companions at a spectral resolution of R ~ 5000 using a silicon echelle grism with a 5 mm pupil diameter at the Lick 3m telescope. These results represent the first scientific observations conducted by the high-resolution silicon grisms, and demonstrate the extremely high dispersing power of silicon-based gratings.

The future of silicon-based grating applications in ground and space-based IR instruments is promising. Silicon immersion gratings will make very high-resolution spectroscopy (R > 100,000) feasible with compact instruments for implementation on large telescopes. Silicon grisms will offer an efficient way to implement low-cost medium to high resolution IR spectroscopy (R ~ 1000 – 50000) through the conversion of existing cameras into spectrometers by locating a grism in the instrument’s pupil location.

Key Words: Silicon grism, Silicon immersion grating, Infrared, Spectroscopy

1. Introduction

Spectroscopy is a major tool for astronomical observation. It is the method used to determine masses, ages, evolutionary histories, chemical composition, star formation rates, redshifts and kinematics of galaxies, AGNs, and quasars. IR spectroscopy, especially moderate to high resolution (R = λ/Δλ ~ 10³ – 10⁵) spectroscopy, plays a critical role in astronomical studies with both ground-based and space-based telescopes. In the NGST report by Mather et al. (2000), medium spectral resolution (R ~ 1500) in the 1-28 µm band is considered to be key to the study.

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of star formation and chemical evolution of young galaxies at high redshifts and also the study of protostars and protoplanetary disks. In the recent Astronomy and Astrophysics decade survey report (McKee et al. 2000), the McKee-Taylor committee identified studying the formation of stars and their planetary systems as well as the birth and evolution of giant and terrestrial planets as one of the key problems to be addressed in this coming decade. High resolution IR spectroscopy is essential for such studies. For instance, IR emission line profiles observed at $R = 10^4$-$10^5$ can tell us the nature of disks, winds, and collapsing gas around protostars. At $R \sim 10^5$, the line profiles of CO and other molecules can provide precise measurements of the physical and chemical properties of young planets (Carr & Najita 1997). IR absorption line profiles of the CO $v=0$-1 band at 4.6 $\mu$m observed at $R \sim 2 \times 10^5$ can address the kinematics, temperature and column density – and thus the mass infall rate – of gas infalling onto the star/disk system.

High resolution astronomical spectrometers usually employ a coarsely ruled diffraction grating, known as an echelle, used in a high order of interference. Under seeing limited conditions, the spectral resolving power is coupled to telescope aperture size,

$$R = \frac{2d}{\theta B} \tan \theta_B = \frac{2}{\Delta \beta} \tan \theta_B,$$

where $\theta_B$ is the angular slit width of the entrance slit, $D$ is the telescope aperture size, $d$ is the diameter of the collimated beam, $\theta_B$ is the blaze angle, and $\Delta \beta$ is the dispersion angle for a spectral resolution element $\Delta \lambda$. For $R = 30,000$ and an R2 echelle ($\tan \theta = 2$), the grating must have a ruled area of $36 \times 72$ cm$^2$ on a 10-m telescope using a 1 arcsec slit. Such gratings and their ancillary optics are becoming so large and expensive that alternative technologies must be sought for the new generation of 8 to 10 m telescopes. This is especially true for the IR where instruments must be operated at cryogenic temperatures. For example, the Keck NIRSPEC, the most powerful existing IR spectrometer with a spectral resolution of $R = 25,000$, has dimensions of $\sim 2 \times 1 \times 1$ m$^3$ and a mass of about 400 kg and has cost more than $3 million and taken 5 years to construct (Mclean et al. 1994; 1998; 2000).

The key approach to reducing instrument size is the use of immersion diffraction gratings instead of conventional reflective gratings. By immersing the grating groove pattern in IR materials, such as silicon with high refractive index ($n = 3.4$ at $2.2 \mu$m), the diffraction grating yields higher dispersion (see Figure 1). The dispersion power of an immersed grating is described as

$$R = \frac{2nd}{\varphi D} \tan \theta_B,$$

where $n$ is the refractive index of the material. Therefore, by employing high index materials such as silicon, one can increase the spectral resolving power of an instrument by more than three times over a conventional reflective spectrograph, or reduce the IR spectrometer linear length by more than a factor of 3 and thus reduce the volume and weight by nearly an order of magnitude for the same spectral resolution. Furthermore, by operating silicon immersion gratings in an anamorphic immersion mode, the increase in spectral resolving power can be up to a factor of $n^2$ or $\sim 12$ times at the Brewster angle (Dekker 1987). This enables very compact, low mass and very high resolution spectrometers for IR spectroscopy.

Silicon immersion grating technology has offered an efficient way to implement low-cost medium to high resolution IR spectroscopy through converting existing cameras into spectrometers by locating a grism in the instrument’s pupil location. A grism is a combination of a diffraction grating and a prism and is designed to have no deviation for the central wavelength as illustrated in Figure 2. The spectral resolving power of a grism is described as
\[ R = \frac{(n-1)}{\Delta \beta} \tan \alpha, \]

where \( \alpha \) is the prism wedge angle. Silicon is an ideal grism material due to its very high refractive index. A silicon grism offers six times the dispersing power of a conventionally made CaF₂ grism \((n \approx 1.4)\), and nearly twice that of a KRS-5 grism \((n \approx 2.4)\), which also can be made using conventional mechanical ruling, but can only be operated in low dispersion orders because it is very difficult to make coarse grooves on these materials by diamond ruling. Compared to reflective gratings, silicon grisms can still offer a factor of 1.2 times the dispersion of a reflective grating of equal length and blaze angle. Therefore, silicon grisms enable medium and high spectral resolution spectroscopy in IR camera systems.

The silicon immersion grating technology based on lithography and anisotropic chemical etching permit the production of gratings with much coarser grooves than are possible with conventional ruling techniques. Currently, no gratings are commercially available with fewer than 23.2 grooves per millimeter (or grooves larger than about 43 \(\mu m\)). Furthermore, the conventional ruling techniques cannot be applied to the relatively brittle silicon. This new grating fabrication approach takes advantage of the crystal structure of silicon to form very sharp V-profiles and very smooth facets through anisotropic etching by certain chemical reagents, e.g. potassium hydroxide (KOH) and tetramethyl ammonium hydroxide (TMAH). For instance, KOH and TMAH attack the silicon (100) planes hundreds of times faster than the (111) planes. The V-shape grating grooves with a 70.52° apex angle are formed once chemicals reach the (111) surfaces as shown in Figure 3. The size of the grooves is determined by the initial grating mask size. Hence, very coarse gratings can be fabricated by careful design of grating masks and control of the chemical etching process. Coarse gratings will allow us to build cross-dispersed spectrometers with large continuous wavelength coverage on 2-D detectors to achieve a multiplex advantage in IR spectroscopy (i.e. increase instantaneous wavelength coverage).

2. Previous Development of Silicon Grating Technology

The concept of immersion gratings was originally proposed in the 1950’s (Hulthen & Neuhaus 1954). In spite of these papers, a patent was issued in 1984 for an immersion grating manufactured using ion milling techniques (Sica 1984). In the early 1990’s, several groups studied the feasibility of making immersion gratings out of silicon substrates using lithography and anisotropic chemical etching techniques (Wiedemann et al. 1993; Kuzmenko, Ciarlo & Stevens 1994; Graf et al. 1994). They chose silicon because it has one of the highest refractive index for potential applications of very high resolution spectroscopy in the near- and mid-IR wavelength regions. The first silicon grating was etched on a thin silicon wafer in the 1970’s (Tsang & Wang 1975). Further studies in 1994 show that the V-shaped, flat-topped groove profiles produced on these wafer gratings with a 25 \(\mu m\) period perform with roughly equal efficiency (~80%) to the 90° ruling profiles of standard (ruled) echelle gratings with the same blaze angle (54.7°) at 10 \(\mu m\) (Graf et al. 1994). In their recent results (Jaffe et al. 1998; Keller et al. 2000), Jaffe and Keller reported about 10% light loss with their wafer gratings in the visible due to diffuse scattering. The best peak grating efficiency approaches ~70% at the same wavelengths. Wiedemann and Jennings (1993) tested a silicon immersion grating and showed that, while they could measure a diffraction pattern in immersion, there was a large amount of light scattered from defects in the groove pattern that severely decreased the diffraction efficiency of the grating, e.g. the measured efficiency in the immersion mode is ~3 times lower than expected. Kuzmenko et al. (1994) have tested a silicon immersion grating and several reflective wafer gratings that they manufactured with photolithography and plasma etching. For the gratings with a 97.5 \(\mu m\) groove spacing they measured a 12% scatter loss in reflection at 0.633 \(\mu m\) that they attributed to intrinsic defects in the silicon crystal lattice and to inconsistencies in the silicon introduced during processing. Later, Kuzmenko & Ciarlo (1998) also found that using high-purity silicon decreases the number of light-scattering defects and improves the quality of chemically etched echelle gratings. Silicon wafer gratings masked with silicon nitride achieved low wavefront distortion (0.07 wave...
RMS at 0.633 \( \mu m \)). The total scatter light with the best gratings shows a ~10% level at 0.633 \( \mu m \) vs. ~3% for the commercially ruled echelle with 23.2 l/mm grooves (or 43 \( \mu m \)).

Other processes have also been proposed for making silicon or germanium immersion gratings. But none of these can produce gratings of a high enough quality for operation in the near-IR. For instance, Ebizuka et al. (2000) demonstrated that diamond turning and grinding methods can possibly produce good quality germanium immersion gratings at 10 \( \mu m \). However, it is very challenging to fabricate gratings useful in the near-IR due to large surface roughness and wavefront distortion of grating facets caused by diamond cutting.

All these previous efforts demonstrate that the new grating techniques are able to produce high quality gratings on thin silicon wafers. However, a true immersion grating, where the etched surface is on the hypotenuse of a prism, requires the processing of thick pieces of silicon and is much more problematic. No one has yet produced a silicon immersion grating of high enough quality to be useful for high-resolution spectroscopy.

Over the last couple of years, several groups, including ours started to develop new processes for making high dispersion grisms out of silicon and germanium (e.g. Käufl et al. 1998; Ge et al. 1999a,b; 2000; 2001; Vitali et al. 2000; Ebizuka et al. 2000). Käufl et al. (1998) successfully demonstrated workable hybrid grisms, made of a germanium prism in optical contact with a spring-loaded thin silicon wafer grating, at ~10 \( \mu m \) and achieved ~60% efficiency. Their attempt to adapt the same process for making a grism working in the near-IR was not successful. No further progress has been reported since. Vitali et al. (2000) proposed to make silicon grisms through wafer bonding techniques. The first results from a bonded grism demonstrate ~33% grating efficiency at 1.7 \( \mu m \), about 20% lower than the expected maximum. The lower efficiency may be due to imperfect bonding. This process may offer an alternative for making silicon grisms and immersion gratings. However, it is very challenging to have perfect bonding between a wafer grating and a prism (see Tong & Gösele 1999 for details).

3. Our Development of Silicon Gratings

3.1. Fabrication of Prototype Silicon Grisms

Our process for fabricating silicon grisms involves the following steps as illustrated in Figure 4. First, an etch-resistant masking layer is grown (silicon dioxide) or deposited (silicon nitride) on the surface of a chemically-mechanically polished silicon disk with flatness of \( \lambda/20 \) at 0.633 \( \mu m \). Photolithography is used to transfer a very precise grating pattern onto the masking layer. The grating pattern is aligned to the silicon crystal axes to within 0.1º, using a splay etching pattern (Kuzmenko & Ciarlo 1994). The grating pattern is transferred to the masking layer using wet chemicals, such as buffered HF (BHF), for dioxide or plasma etching for nitride. Once the silicon is exposed, immersion in aqueous KOH etches the material down to the (111) crystal planes. These planes form the grating facets. After the etching is finished, the mask layer was removed in BHF. The gratings are cut in prism shape and polished at the entrance surface to rms flatness of \( \lambda/10 \) at 0.633 \( \mu m \). Finally, both the entrance and the grating surfaces of finished silicon grisms are deposited with a ~500 nm layer of silicon nitride as an anti-reflection (AR) coating.

Previous to our first attempts at fabricating silicon grisms in 1999, we etched 25 sets of silicon gratings on wafers in order to find the best process. Each wafer has 20 gratings with an area of 10×10 mm\(^2\). We used three different etch mask materials: silicon dioxide, silicon nitride and chromium/gold (Si\(_3\)N\(_4\)/Au/Cr) on top of the thin wafers. For the silicon nitride and oxide layers typical thickness is about 100 nm and 600 nm, respectively. For the chromium/gold mask, we deposited a ~100 nm layer of silicon nitride in the bottom, ~10 nm chromium layer in the middle and ~
100 nm Gold layer on the top. The advantage of using a nitride mask layer is its negligible etch rate in aqueous KOH helps preserve the masking pattern accuracy. However, the standard plasma process for nitride etching has low selectivity between silicon and the nitride. The nonuniform overetching, to insure the masking layer being completely etched through, causes variations in the position of the facets producing wavefront errors that degrade spectral resolution. In addition, since all the industrial standard plasma etchers are designed for processing thin wafers, it is challenging to etch very thick silicon substrates with plasma, especially when it comes to controlling etch speed. The advantage of using a silicon oxide or chromium/gold mask is that we can process silicon substrates without a dry etching process (plasma etching). However, the grating pattern cannot be preserved as sharply as that produced by dry etching, which can result in larger wavefront errors and higher scattered light than nitride mask gratings.

These wafer grating experiments demonstrate that better gratings are made with plasma etched silicon nitride masks than the wet etched masks (oxide and Cr/Au masks), similar to what has been found in previous study (Kuzmenko & Ciarlo 1998). The optimal etching process is the one with a ∼ 100 nm thick silicon nitride mask and plasma etching (the etch gas with a 10:1 ratio of CF_4 to O_2 at 200 mTorr), which provides the lowest wavefront distortion, grating surface roughness and minimal grating defects (e.g. less than 1 visible defect per cm^2 and no scallops under a 100X microscope). The scallops, caused by local mask failure during the anisotropic etching, contribute to diffuse scatter on large scale. We then applied this technique to a silicon disk (4 cm thick and 10 cm in diameter) and successfully made 9 gratings in Fig. 5). They blaze angle. For errors are 0.024, reflection at performance in by a commercial 1998. This better than the & Ciarlo (1998). 2.104 µm (Figure the UC Berkeley a diffraction- with a 0.15 arcsec entrance slit. This camera was diffraction-limited designed to provide high spatial imaging at the Lick 3m with the LLNL laser guide star adaptive optics system (Lloyd et al. 2000).

The great challenge for fabricating these prototype grisms is to control grating facet roughness, which causes diffuse scattered light in spectra, reduces grating efficiency and increases measurement uncertainty. We found that gratings on times roughness with wafer process. plasma rpm vs. mask and directly layer on variable surfaces...
and defects. The rough grating facet and defects result in relatively high integrated scattered light in our prototype grisms, measured at ~34% in the K band (Figure 8). The scatter is distributed over a large area and the resulting grism peak efficiency is about 36%.

3.2. Development of Second Generation Silicon Grisms

In 2000, we ran more experiments with wafer gratings and explored new techniques to reduce grating surface roughness with the KOH etching process. We experimented with four etch masks: silicon dioxide, nitride, Cr/Au and a new hybrid mask with silicon nitride and silicon dioxide layers. This hybrid mask with a thin oxide layer (~250 nm thickness) underneath a thin nitride layer (~100 nm thickness) eliminates silicon subsurface damages due to overetching of plasma gas since the oxide layer provides a buffered layer. Though the oxide etch rate is somewhat faster than nitride in our plasma etcher, the plasma etching can be stopped after the plasma finishes etching the nitride layer. The remaining oxide layer was finished by BHF. The physical and chemical conditions for etching these wafer gratings were the same. The etched gratings were evaluated by inspecting defects with a microscope, measuring rms roughness with a stylus profilometer, and measuring rms wavefront errors with a Zygo interferometer.

Table 1. summarizes rms roughness and wavefront distortion from the measurements.

<table>
<thead>
<tr>
<th>Grating mask</th>
<th>Averaged rms roughness (nm)</th>
<th>Lowest rms roughness (nm)</th>
<th>Averaged rms wavefront error (waves)</th>
<th>The lowest rms wavefront error (waves)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry etched nitride</td>
<td>33.6</td>
<td>16.2</td>
<td>0.126</td>
<td>0.103</td>
</tr>
<tr>
<td>Wet etched nitride (H3PO4)</td>
<td>25.4</td>
<td>16.2</td>
<td>0.103</td>
<td>0.077</td>
</tr>
<tr>
<td>Wet etched oxide</td>
<td>38.2</td>
<td>16.7</td>
<td>0.049</td>
<td>0.021</td>
</tr>
<tr>
<td>Hybrid oxide/nitride</td>
<td>30.3</td>
<td>8.4</td>
<td>0.035</td>
<td>0.028</td>
</tr>
</tbody>
</table>

Table 1. Optical properties of silicon gratings etched under the same physical and chemical conditions of KOH but with different etch masks.

It is evident that the hybrid mask gratings provide the lowest wavefront error and roughness. This is because the hybrid mask allows us to take advantage of the very sharp mask pattern provided by plasma etching and also smooth etched grating surfaces with the wet etched oxide mask. The best rms roughness over-etched gratings for each of the four masks is within 20 nm, which has been significantly improved from the previous ~50 nm rms roughness.

We applied this new hybrid mask technique in fabricating two large gratings with 38×81 mm² etched area, a 54.7° blaze angle and 75 µm grooves (or 13.3 l/mm groove frequency) on two silicon substrates with 100 mm diameter and 10 mm thickness. The grating mask was interferometrically ruled to minimize grating ghosts (Kuzmenko & Ciarlo 1998). Figure 9 shows one of the etched gratings. The other grating was cut into several 20×40×10 mm³ cubes and one of them with 0.028 wave rms wavefront error at 0.633 µm was polished to make a silicon grism. This grism was tested in the IRCAL camera in September 2000, showing ~8% integrated scatter in the K band (Figure 10), a factor of 4 times less than our prototype grisms shown in Figure 8. Unlike the prototype grisms shown in Figure 8, most of the scattered light is concentrated within 3 times the image size. This is probably due to elimination of large-scale defects (~100 µm) in the new grating surface with the hybrid mask. This kind of scattered light does not affect precision for spectroscopic
measurements as long as the spectral order separation is much larger than 3 times the image size. It only affects detection sensitivity. The integrated scatters in the H and J bands, which show similar intensity distribution in the spatial direction as in the K band, are 12% and 14%, respectively.

Due to the decrease in total scattered light, the grating peak efficiency has been increased to 45% in the 70th echelle order (Figure 11, Ge et al. 2001). This is comparable to that of medium resolution KRS-5 grisms commercially available for use only in the first grating order, (~45% efficiency measured with the GEMINI NIRI near-IR camera, Simons 2001, private communication).

3.3. Demonstration of Scientific Capability at Observatories

The field demonstration of silicon grisms was carried out in the IRCAL camera with a 5 mm pupil diameter at the Lick 3m telescope in 1999 and 2000 and also in a GSFC 1024x1024 InSb camera at the APO 3.5m telescope in March 2000. These tests demonstrate a diffraction-limited spectral resolution at 2.1 µm (Fig. 7). The grism made through the hybrid nitride and dioxide mask in early 2000 was further used for initial scientific observations at the Lick 3m in September 2000 and complete K band spectra were obtained for six young stellar objects (YSOs). The initial results were presented in the American Astronomical Society meeting at San Diego in January 2001 (Ge et al. 2000). Figure 12 shows a cross-dispersed silicon echelle grism spectrum of the T Tauri N companion. Brγ emission lines have been found associated with both T Tauri N and S components (Figure 13). These observations demonstrate scientific capabilities for silicon grisms. They also demonstrate another advantage of silicon grisms: enabling large simultaneous wavelength coverage through operation in high dispersion orders. This is achieved by silicon grisms operated in 65-80 orders with very coarse grooves (~ 13 l/mm, a factor of two coarser than commercially available ones). With a low dispersion cross-disperser, ~16 cross-dispersed orders of spectra is packed on the 256×256 PICNIC array in the IRCAL camera. Hence, silicon grisms or immersion gratings allow large, instantaneous continuous wavelength coverage on 2-D detectors of any size. Observation efficiency can be correspondingly increased.

3.4. Development of Third Generation Silicon Grisms

In 2001, we developed one of the most promising etching processes: using TMAH for chemical etching instead of the traditional KOH. This process allows us to use wet etched masks instead of the previous masks involving plasma etching, yet still provides the same wavefront control and even better grating facet roughness control. In previous efforts by other researchers (e.g. Jaffe et al. 1998; Kuzmenko & Ciarlo 1998), very thick dioxide layers (~ 1000 nm) had to be used. Wet etched grating masks on thick dioxide layers do not yield grating patterns as sharp as plasma etched ones due to the isotropic etching nature of chemicals in the dioxide layer. This undercutting of chemical etching causes wavefront error and surface roughness as demonstrated in Table 1. This situation can be significantly improved by this new TMAH etching process. The main reason to have very thick dioxide in previous efforts is that KOH attacks the dioxide layer during the anisotropic etching of the silicon crystal and only a dioxide etch mask with thick layers can
survive the KOH etching and preserve grating patterns in silicon. Unlike KOH, TMAH does not attack dioxide during anisotropic etching. One can therefore use a thinner dioxide layer. The sharp grating pattern in the photoresist layer can be easily transferred to the thin dioxide layer without significant degradation of mask sharpness as with the thick dioxide mask. All the advantages of a sharp etch mask, such as lower wavefront distortion, lower surface roughness and fewer defects, previously achieved with dry etching (plasma etching) can be maintained with the new wet etch mask.

Some preliminary studies of this new mask etching process have been conducted recently at Penn State and demonstrated ~10 nm rms surface roughness on the etched wafer gratings. A scanning electron microscope (SEM) picture of one of the etched gratings is shown in Figure 14, indicating very smooth grating surfaces. This grating was further evaluated at 0.633 µm by an optical spectrograph for measuring its global optical properties. The total measured integrated scatter at 0.633 µm is less than 1% as shown in Figure 15. This level of scatter is already a factor of three times better than that of a commercial 23.2 l/mm echelle grating measured by our collaborators, Kuzmenko & Ciarlo in 1998. This scatter level corresponds to <1% scatter light in the near-IR (1.2-2.4 µm). The wavefront error with the first TMAH etched grating was measured with a Zygo interferometer and shows about 0.1 waves at 0.633 µm over 10×10 mm² etched grating area. The wavefront errors for the newly etched gratings are being evaluated with a newly constructed interferometer at Penn State and will be reported in future papers (Ge et al. 2001).

New experiments testing the addition of ammonium persulfate (AP, an oxidizer) in the TMAH etching process have demonstrated that this new technique can further reduce surface roughness by eliminating hillocks formed during chemical etching. Recent studies by other researchers found that this new process can help to maintain a constant etch rate and reduce hillocks on the etched surfaces (e.g. Paranjape et al. 2000; Pandy et al. 1998; Sullivan et al. 2000). These hillocks varying in size may contribute significantly to diffuse scatter in the etched gratings. As of this publication, we have performed several experiments with adding AP to the TMAH etching process and preliminary results indicate that rms roughness has been reduced by ~25% over a control sample of TMAH etching without AP. Figure 16 shows the SEM profiles of etched grating bottoms with and without AP during the etching. Without AP, many hillocks were formed on the sidewalls and bottoms of the gratings.

4. Discussion and Future Plan

![Figure 16. SEM profiles of silicon wafer gratings etched with TMAH without (a) and with (b) ammonium persulfate. Hillocks can be clearly seen in the picture without AP.](image)

Our previous developments indicate that a process for fabricating gratings on silicon substrates with chemical etching involves a number of exacting steps, each of which must be successfully completed in order to produce a useful device. The typical steps are illustrated in Figure 4. Among these steps, several play critical roles, such as
printing of etch masks and chemical anisotropic etching. Table 1 illustrates that a good etch mask can lead to low wavefront errors and surface roughness, which are required for providing diffraction-limited spectral resolving power and high efficiency of etched gratings. In our development, we found that different physical and chemical etching conditions in both KOH and TMAH can result in significantly different quality gratings in the end. Though the details in each of the steps need to be further investigated, our current developed techniques should allow us to fabricate gratings on small area of silicon substrates with a quality high enough to produce diffraction-limited silicon grisms for IR spectroscopy as demonstrated with the Lick scientific observations. However, in applying our techniques to make large silicon grisms, further technology development is necessary in order to reduce scatter light and increase grating efficiency. To make silicon immersion gratings, we also need to improve wavefront quality over a large area. For instance, in our first etched silicon gratings on a thick silicon disk with 10 cm diameter and 4 cm thickness, there was a factor of 7 variations in wavefront error between the best (0.024 wave at 0.633 μm) and the worst gratings (0.178 wave at 0.633 μm) that were all printed with the same lithography step. In summary, further development in reducing surface roughness and wavefront distortion is crucial to produce large, high quality silicon-based gratings for IR spectroscopy.

The major processes for making silicon immersion gratings and grisms are the same. The main difference is the requirement of the grating surface quality (wavefront distortion and grating facet roughness), the prism shape and the coatings for the grating surface. The requirement of the grating surface quality for an immersion grating is about a factor of $2\pi/(n-1) \sim 3$ times tighter than that for a grism. The prism shape cut out of the silicon disk for a grism is different from that for an immersion grating in order to optimize the grating throughput due to the special $70.5^\circ$ groove angle formed by the silicon (111) crystal surfaces instead of $90^\circ$. Details will be discussed in a later paper (Ge et al. 2001 in preparation). For an immersion grating, a coating of gold on the grating facets completes the process. For a grism, an AR coating on the grating facets completes the process.

The great technical challenge for etching large silicon gratings for near-IR spectroscopy is to further reduce the grating facet roughness. For example, in order to make a silicon grism to work at 1.2 μm with < 1% total integrated scatter, the rms surface roughness of ~ 0.037 nm is required for silicon grisms based on a scalar scattering theory (Bennett & Mattsson, 1989). For an immersion grating, a rms surface roughness of < 3 nm is required for < 1% scatter at the shortest operation wavelength of 1.2 μm. We have achieved the rms roughness of ~ 10 nm with the hybrid Si$_3$N$_4$/SiO$_2$ etch mask + KOH and SiO$_2$ etch mask + TMAH processes. Further reduction in surface roughness is required to achieve < 1% scatter over the near-IR region.

Near-IR operation also requires control of grating wavefront distortion over large areas. For a silicon grism to have diffraction-limited performance at 1.2 μm, an rms wavefront error < 0.086 μm is required. This wavefront quality has already been achieved with our prototype grisms over 10x10 mm$^2$ etched area. In order to reduce wavefront distortion over larger areas, very flat (λ/20 rms) silicon substrates are needed to start with and very tight control over lithography and etching processes is also necessary.

Silicon has good transmission over 5-10 μm at very low temperatures. Silicon based gratings will have potential applications for space based instruments since the atmosphere is quite opaque over most part of the wavelengths. In these mid IR wavelengths, the requirement for grating surface roughness is less stringent than that in the near-IR. For a grism to have less than 1% total integrated scatter at 5 μm wavelength an rms roughness of only 34 nm is required. This roughness requirement is already within the scope of our current etching technology. In order to operate the immersion gratings at the same wavelengths, the surface roughness of ~ 11 nm is required to have < 1% total scatter light level. This level of surface roughness has already been achieved with our techniques.

Mid-IR operation also reduces the requirement on wavefront distortion at the grating surface. The tightest constraint will be at 5 μm, the shortest wavelength for the mid-IR instruments. For a grism, a wavefront distortion of $\lambda/14$ is required to provide a diffraction-limited spectral resolution. The worst wavefront distortion in our prototype grisms is $\lambda/18$ at 5 μm. Therefore, the wavefront error in our previous process has already met the requirement for mid-IR diffraction-limited operation of grisms. For the silicon immersion grating, the best wavefront quality we have achieved should allow diffraction-limited performance. Therefore, our current techniques should be able to produce high quality and large silicon immersion gratings and grisms for mid-IR spectroscopic instruments.
We plan on following work over the next few years in order to make our grating technology ready for producing high quality and large silicon based gratings for IR spectroscopy.

### 4.1. Reduction of Surface Roughness

Effort will be placed into understanding the dynamics of the chemical etching process in order to determine and remedy the cause of the surface roughness. Our main goal is to reduce etched grating surface roughness to ~ 3 nm from the ~ 10-50 nm obtained with our current techniques. The resulting total scattered light level is < 1% in immersion mode at wavelengths short of 1.2 $\mu$m.

A primary cause of surface pits during chemical etching is due to the presence of defects in the silicon. These defects can be created during the crystal growth process or develop during a later processing step. The presence of residual oxygen in the crystal is the usual culprit. When the silicon is then heated, oxygen atoms migrate to point defects in the crystal structure and agglomerate to form macrodefects. We will experiment with specially grown silicon (float zone, FZ) with very low oxygen content and also attempt to process conventional silicon such as Czechralski (CZ) silicon with high oxygen content at reduced temperatures. Metallic impurities, which have very high mobilities in silicon, can cause similar problems. Special cleaning procedures, e.g. RCA for pre-etch cleaning, developed in the microelectronics industry and routinely practiced in the NanoFab, will be used to remove metallic impurities from surfaces. This will be carried out before any high temperature steps in the processing and before chemical etching. Vacancies and interstitials are a final form of defect. They are generated at high temperatures during crystal growth and can fail to recombine during the rapid cooling that follows. High temperature annealing in an inert atmosphere, followed by a slow cool down should reduce the density of these inherent point defects.

The final important step in our silicon grating fabrication is the actual anisotropic etching of the silicon in hot KOH or TMAH. Although we have achieved a fair amount of success in our past efforts, there are many variables yet to be studied. Some of the more obvious variables are temperature, concentration, agitation including ultrasonic energy and the use of organic additives. We already have extensive experience with these issues. One result we found is that the eventual grating surface roughness is significantly contributed by this step. KOH or TMAH chemical etching produces small hydrogen bubbles on the etched silicon surface. These act in effect as tiny transitory etch masks, the net effect of which is to increase the roughness of the etched surface. This roughness results in excess scattering and causes stray light problems in spectroscopic instruments. Ultrasonic agitation will be used during the KOH or TMAH chemical etching to break free the hydrogen bubbles and reduce the roughness of the etched grating facets. We plan to apply ultra-sonic agitation in the chemical etching process to further reduce the surface roughness.

We also plan to conduct post etch smoothing techniques. A previous study indicates that wet oxidation ($1100^\circ$ C for 90 minutes) of finished silicon gratings followed by an HF etch to remove the oxide can further reduce rms surface roughness (Kwa et al. 1995). After two oxidation and etching steps, roughness was reduced by a factor of two to three in this previous effort.

### 4.2. Development of Fine Groove Silicon Gratings

Our previous work has produced coarse grooves for echelle gratings using contact lithography. There is a strong need for fine pitch (d ~ 1-5 $\mu$m) gratings operating in low spectral orders with higher grating efficiency such as multi-object spectroscopy and medium resolution single-object spectroscopy in the near-IR. Unless extraordinary efforts are made, contact lithography is only useful for printing features greater than 5 $\mu$m. We plan to develop a new lithography procedure with an available 248nm Nikon projection stepper at NanoFab that can produce the current minimum feature size of ~ 0.2 $\mu$m. However, this stepper has a relatively small field of view of 30x30 mm$^2$. To fabricate gratings with larger areas requires translation of the substrate and printing additional patterns. It is very challenging to control the relative positioning of these patterns with the required accuracy (<0.1 $\mu$m) in order to maintain the proper phase relation. Additional study is necessary to find solutions for accurate grating pattern stitching.

Another effective approach is interference lithography, which involves using the interference pattern from a UV laser to form finely spaced straight lines in photoresist. This is essentially the same procedure used to make holographic gratings. By appropriate choice of laser exposure, one can produce an evenly spaced set of narrow lines (period > 0.1 $\mu$m) in the resist. High efficiency X-ray gratings with very shallow blaze angles have been produced
by this technique (Franke et al. 1997). We plan to use an interference lithography setup at LLNL for printing fine period grating patterns if the grating pattern stitching technique fails to meet the requirement.

4.3. Modeling immersion grating efficiency and AR coating performance

We have used a commercial grating vector calculation code, GSOLVER, to study the grating efficiency and blaze functions at different IR wavelengths. Figure 17 shows the calculated grating efficiency of a silicon grism with a 10° blaze and 5.35 μm grooves in the first and second dispersion orders. It is designed for the NGST prototype near-IR MOS (RIVMOS) to provide a moderate spectral resolution $R \approx 2,000$ for multi-object spectroscopy in the K band. We can achieve 82% efficiency at the peak of the blaze for a silicon grism with a quarter-wave silicon nitride coating. Therefore, this kind of grism would allow very efficient observations in the near-IR.

In order to achieve its potential high efficiency $\sim 80\%$, a silicon grism will require an antireflection coating. Although the technology is well developed for smooth surfaces like windows and lenses, producing a uniform coating on the angled surfaces of a grating is far from routine. We plan to continue to explore techniques other than the standard sputtering or e-beam evaporation for coating the grating. In particular, chemical vapor deposition (CVD) offers the possibility of achieving a conformal coating of high quality. Research is required to determine the best materials and processes.

We plan to do further modeling of silicon based gratings in order to understand their grating efficiency and polarization properties to guide our development. This modeling is especially important for understanding grating performance under low order operation since scalar calculations break down and a full vector computation must be made. This vector calculation can be done by this GSOLVER code.

4.4. Grating Properties Characterization

Characterization of etched silicon gratings is crucial to our development. The grating surface characterization will continue to be performed in the Penn State NanoFab and Material Research Institute to study defects and surface roughness. The information will provide critical diagnostics in our process. The state-of-the-art facilities involved in characterization include: Optical microscopes for finding any defects, a SEM and FESEM for a very detailed knowledge of any groove defects such as shingles, scallops and hillocks, a stylus profilometer for measuring the surface roughness over a large etched area, an ellipsometer for measuring the mask layer thickness, an atomic force microscope (AFM) for measuring surface roughness over very small areas, and an Auger Spectrometer for identifying the surface contaminants. These tools should allow us to evaluate the etched grating quality including rms roughnesses above 1 nm and rms wavefront errors above $\lambda/50$.

The characterization continues to be evaluated in the astronomy department of Penn State. The grating optical properties include wavefront distortion, grating efficiency, resolving power, ghost images, scatter and blaze function. The facilities include a Michelson interferometer for measuring wavefront distortion, an optical spectrometer for measuring grating efficiency, resolving power, ghost, scatter and blaze function. There is also a near-IR camera for evaluating grating optical performance in the near-IR.

We will first test the gratings in reflection mode using a visible HeNe laser, continuum lamps and a CCD camera to evaluate their optical performance. The grisms will be further evaluated in transmission mode in the infrared with the IR camera, an IR laser, emission line lamps, and continuum lamps for testing the performance in a cryogenic environment. The immersion gratings will also be evaluated in immersion mode with the IR camera at room temperature. Grating resolving power, efficiency, blaze function, scattered light level and ghost images at 1 – 2.5 μm will also be measured.
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