Progress in the Development of Critical-Angle Transmission (CAT) Gratings

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Why High-Resolution Soft X-Ray Spectroscopy?

• Science:
  – Detection of missing baryons in the Warm-Hot Intergalactic Medium (WHIM).
  – Structure of galactic haloes and relation to dark matter.
  – Evolution of young stars.
  – Composition of Interstellar Medium (ISM)/gas & dust, and its significance, from nucleosynthesis to planet formation.

• Measurement Technique:
  – Resolve the absorption and emission signatures of atomic ions in the soft x-ray band (~ 0.3 – 1 keV) which reveal temperatures, compositions, and dynamics of the involved plasmas.
High-Resolution Soft X-Ray Spectroscopy - how?

Diffraction Gratings!

![Graph showing resolving power vs. energy for different instruments](image-url)
Diffraction Gratings for X-ray and EUV Spectroscopy

Transmission Gratings
- 1st order
- Relaxed alignment & surface flatness tolerance
- Low mass
- Low diffraction efficiency (absorption, etc.)

$$m\lambda = p\left(\sin \alpha + \sin \beta_m\right)$$

Blazed Reflection Gratings
- Grating normal
- Facet normal
- Requires precise alignment, flat & smooth surface
- High diffraction efficiency
- High mass

**Grating Equation**

- $m$: diffraction order
- $\lambda$: wavelength
- $p$: grating period
- $\alpha$: incident angle, $\beta_m$: diffracted angle
Critical-Angle Transmission Grating Spectrometer: Blazed diffraction enabled by CAT gratings

- CAT grating combines advantages of transmission gratings (relaxed alignment, low weight) with high efficiency of blazed reflection gratings.
- Blazing achieved via reflection from grating bar sidewalls at graze angles below the critical angle for total external reflection.
- High energy x rays undergo minimal absorption and contribute to effective area at focus.

CAT grating principle

Grating equation:

\[ m \lambda = p (\sin(\theta) + \sin(\beta_m)), \]

\[ m = \text{diffraction order} \]

**Blazing:** \( \beta_m \approx \theta \)

**High reflectivity:**
\[ \theta < \theta_c = \text{critical angle of total external reflection} \]

Strawman:
- Silicon grating, \( \theta = 1.5^\circ \)
- \( p = 200 \text{ nm} \)
- \( b = 40 \text{ nm} \)
- \( d = 6 \text{ \mu m} \)
- aspect ratio \( d/b = 150 \)

Efficiency comparison with Chandra gratings
**Chandra Heritage:**
High Energy Transmission Grating Spectrometer (HETGS)

**Chandra Telescope**

**HETGS Instrument (MIT)**

Rowland Torus Transmission Grating Geometry and CCD Readout Array

Space Nanotechnology Laboratory
Heilmann *et al.*, PCOS XRSAG
CAT Grating Development
Critical-Angle Transmission (CAT) Grating Spectrometer Concept

- **Optical Design:**
  - Wolter I telescope mirrors.
  - Diffraction gratings in converging beam just aft of mirrors.
  - Gratings, camera, and focus share same Rowland torus.
  - Blazed gratings; only orders on one side are utilized.
  - Only fraction of mirrors is covered: “sub-aperturing”.

**Advantages:**
- low mass
- relaxed alignment & figure tolerances
- high diffraction efficiency
- up to 10X dispersion of Chandra HETGS
- no positive orders (i.e., smaller detector)
Post-IXO CATXGS
Mission Concepts
(2011 NASA RFI Responses)

- AXSIO (large size, microcalorimeter, gratings + CCDs)
- AEGIS (medium size, gratings + CCDs)
- N-XGS (medium size, gratings + CCDs)
- SMART-X (large size, microcalorimeter, active pixel sensor, gratings + CCDs)
Grating Membrane “Unit Cell”
(etched from silicon-on-insulator (SOI) wafer)

(not to scale)

Level 1 supports (5-10 micron period)

Level 2 support

<111> planes

CAT grating bars (200 nm period)

~ 500 um (handle layer)

~ 4 - 6 um (device layer)

~ 500 nm (SiO2 layer)
X-ray data analysis of diffraction efficiency from wet-etched CAT gratings

Lines – theory; points – measurement/experimental data
2 different samples
(see Heilmann et al., Appl. Opt. 50, 1364-1373 (2011) for details)
CAT grating fabrication and testing (past)

- Monolithic silicon structure with integrated L1 support bars
- 200 nm period
- achieved IXO design goal of 6 μm tall, 40 nm wide grating bars
- wet etch in KOH gives smooth side walls

However:
- Level 1 supports broaden & rob area
- Small gratings without Level 2 support mesh

CAT grating schematic

Scanning electron micrograph of cleaved cross section of 200 nm-period CAT grating
Si crystal-lattice-independent anisotropic etching: Deep Reactive-Ion Etching (DRIE)

- alternates between isotropic SF$_6$ etch step and C$_4$F$_8$ passivation step
- scalloping can be reduced through faster switching (newer DRIE tools)
- need high-aspect ratio DRIE mask
- expect to still need KOH “polishing” step after DRIE to smooth out remaining scalloping/roughness
Process Flow

- Silicon-on-Insulator (SOI) Wafer
- Back side: Millimeter-scale hexagon
- Front side: 200 nm-period CAT grating plus 5 µm-period cross support grating (not shown)

Prepare SOI Wafer

- Thermal SiO$_2$ (400 nm)
- Device Silicon (4,000 nm)
- Buried SiO$_2$ (500 nm)
- Bulk Silicon (500 µm)
- Thermal SiO$_2$ (400 nm)
- PECVD SiO$_2$ (4 µm)

Pattern Mask Front & Back Side

- Device Silicon (4,000 nm)
- Buried SiO$_2$ (500 nm)
- Bulk Silicon (500 µm)
Process Flow

Deep Reactive-Ion etch (DRIE) front side and stop on buried SiO₂
Process Flow

Fill front side with photoresist

Bulk Silicon (500 µm)
Buried SiO₂ (500 nm)
Photoresist
Buried SiO₂, 200 nm pitch grating
Process Flow

Flip over, bond to carrier, DRIE backside

SEM Image of Sample From Bottom

1 mm-Hexagon Support
Large area (31x31mm²) gratings with two levels of support
Freestanding Grating

Etch buried SiO₂, free wafer from carrier, clean photoresist filling, critical-point dry and ash to finish.

5 μm L1 Support

200 nm pitch CAT grating bars
Large Area Grating

Photograph of grating film next to US Quarter
DRIE gives narrow L1 supports with vertical sidewalls (large open area for CAT gratings), but the CAT grating bar sidewalls are rough (scalloping).

**SOLUTION:** Etch in KOH after DRIE.

(a) SEM of cleaved 200 nm-period CAT grating after DRIE (sidewall roughness > 20 nm).

(b) SEM of cleaved 200 nm-period CAT grating after DRIE and 20 min. KOH polish. Roughness is below SEM resolution (< 5 nm).

(c) Same as (a). Observe bowed grating bar profiles with narrow waists.

(d) Same as (b). Observe nearly uniform and straight grating bars.

**“Polishing” of Sidewalls (I)**
“Polishing” of Sidewalls (II)

Etch in KOH after DRIE.

(a) SEM of cleaved 200 nm-period CAT grating after DRIE (*new improved recipe*). Slightly narrow waists.

(b) Same as (a). Sidewall roughness ~ 10 nm.

(c) SEM of cleaved 200 nm-period CAT grating after DRIE and 43 min. KOH polish. Straight sidewalls; thin, slightly trapezoidal grating bar profile.

(d) Same as (c). Roughness ~ 1 nm as measured by AFM.
Advanced DRIE Tool

• Currently using STS Pegasus tool at UMich
  - slow logistics, tool shared by many users

• Evaluating three advanced DRIE tool vendors
  - first round of samples under way
  - first results very encouraging

• Expect to install new tool by fall
Level 2 support mesh and facet frames

- Fabricating Si L2 support mesh membranes with ~90% open area
- Bonding (epoxy, reactive bonding) of membranes to different facet frame materials (Invar, H-Invar, Hexoloy SiC, etc.)
- Investigate mechanical/elastic properties of bonded facets, (shake & bake) compare to finite element models, optimize design
Summary

- Previously fabricated small CAT grating prototypes (KOH wet etch) with small throughput and demonstrated good agreement with predicted x-ray performance.
- Fabricated large-area, high throughput CAT grating membranes with full structural complexity of L1 and L2 supports via DRIE.
- Polished DRIE’d grating bar sidewalls to ~ 1 nm roughness on bulk Si samples.
- Need to
  - Integrate polishing step into fabrication flow for SOI wafers.
  - X-ray test individual grating and grating array.
- Explore L2 mesh designs, frame bonding, facet properties
- Finite-element modeling and “shake & bake” tests for structure optimization (L1 & L2 supports, facet frames)
# X-Ray Grating Spectrometer Comparison

<table>
<thead>
<tr>
<th></th>
<th>Mirror PSF [arcsec]</th>
<th>Grating PSF [arcsec]</th>
<th>Dispersion angle [deg]</th>
<th>$R(\lambda/\Delta\lambda)$ @ 15 A</th>
<th>Mass [kg]</th>
</tr>
</thead>
<tbody>
<tr>
<td>RGS (blazed reflection)</td>
<td>~ 12</td>
<td>~ 2-3</td>
<td>~ 4.8</td>
<td>200-250</td>
<td>2 x 58</td>
</tr>
<tr>
<td>HETG$^1$ (transmission)</td>
<td>~ 0.5</td>
<td>irrelevant</td>
<td>~ 0.43</td>
<td>1500</td>
<td>10.41</td>
</tr>
<tr>
<td>CATGS$^{1,2}$ (blazed transmission)</td>
<td>~ 5 - 10</td>
<td>irrelevant</td>
<td>~ 3 - 4</td>
<td>3000 – 5000$^3$</td>
<td>~ 5 - 10</td>
</tr>
</tbody>
</table>

$^1$transmits non-diffracted and non-absorbed x rays to telescope focus  
$^2$highest diffraction efficiency  
$^3$with sub-aperturing