Development of lightweight blazed transmission gratings and large-area soft x-ray spectrographs

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ABSTRACT

Large area, high resolving power spectroscopy in the soft x-ray band can only be achieved with a state-of-the-art diffraction grating spectrometer, comprised of large collecting-area focusing optics with a narrow point spread function, large-area high-resolving power diffraction gratings, and small pixel, order sorting x-ray detectors. Recently developed critical-angle transmission (CAT) gratings combine the advantages of transmission gratings (low mass, relaxed figure and alignment tolerances) and blazed reflection gratings (high broad band diffraction efficiency, utilization of higher diffraction orders). Several new mission concepts containing CAT grating based spectrometers (AEGIS, AXSIO, SMART-X) promise to deliver unprecedented order-of-magnitude improvements in soft x-ray spectroscopy figures of merit related to the detection and characterization of emission and absorption lines, thereby addressing high-priority questions identified in the Astro2010 Decadal Survey "New Worlds New Horizons". We review the current status of CAT grating fabrication, present recent fabrication results, and describe our plans and technology development roadmap for the coming year and beyond.

Keywords: x-ray optics, critical-angle transmission grating, x-ray spectroscopy, blazed transmission grating, soft x-ray, silicon-on-insulator, deep reactive-ion etching

1. INTRODUCTION

Unlike in the optical and near-optical wavelength bands, high-resolution spectroscopy can be performed efficiently over a whole decade of soft x-ray wavelengths ($\sim$ 6 - 60 Å or $\sim$ 0.2 - 2 keV) with grazing incidence mirrors, diffraction gratings, and CCD detectors of a single design. This wavelength band provides source diagnostics from the characteristic lines of ionized carbon, nitrogen, oxygen, neon and iron, which are central to studies of the Warm Hot Intergalactic Medium, the search for the missing baryons, the study of the outflows of supermassive black holes and the properties of galaxy halos, teaching us about the evolution of large scale structure and cosmic feedback. Soft x-ray spectroscopy of individual stars can help to reveal the effects of rotation, magnetic fields and stellar winds in stellar coronae. More details and additional science cases can be found in Bautz *et al.*[1] and references therein.

Many of these topics were identified as high-priority science questions in the Astro2010 Decadal Survey "New Worlds New Horizons" (NWNH)[2] to be addressed by the International X-ray Observatory (IXO).[3] IXO addressed soft x-ray spectroscopy science questions with a a large-area ($>$ 1000 cm^2), high resolving power ($R = \lambda/\Delta\lambda > 3000$) x-ray grating spectrometer (XGS)[4,5] with more than an order of magnitude larger effective area and at least three times larger resolving power than existing observatory class x-ray spectrometers.[6,7] Soon after the publication of NWNH new realities led to the cancellation of IXO and raised the question of how to fill the gap left in its demise. In response NASA issued a Request For Information (RFI)[8] to solicit "concepts that meet some or all of the scientific objectives of the International X-ray Observatory" at reduced cost.

Among the submitted RFI responses a stand-alone CAT grating spectrometer mission named AEGIS (Astrophysics Experiment for Grating and Imaging Spectroscopy)[1,9] promises the most progress over the state-of-the-art in soft x-ray spectroscopy, exceeding IXO XGS requirements at an estimated cost of < $800M. The low cost is partially due to the properties of CAT gratings: An array of CAT gratings can cover a given mirror area at a fraction of the mass required for a reflection grating array of the same area,[9] thereby allowing increased

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mirror and grating area without increasing total mass; CAT gratings are transmission gratings and therefore significantly less sensitive to misalignment, requiring less mass for alignment and supporting structures; CAT gratings are passively temperature controlled due to their thinness and proximity to the telescope mirrors,[4] again reducing mass and power requirements; and CAT gratings are much more efficient than previous generation x-ray transmission gratings,[10] reducing the mirror area required to achieve a goal effective area.

An X-ray Mission Concepts Study Team was formed to analyze all RFI responses, and to develop a small set of notional missions that meet a set of performance metrics to address key IXO science questions. Due to limited resources only a single notional x-ray grating mission concept was developed (N-XGS), focusing on an off-plane grating design (similar to WHIMex),[5] under the condition that the derived mission parameters can accommodate both a CAT grating and an off-plane grating spectrometer design.[9,11] Since the N-XGS was a compromise design it is not surprising that it performs badly (less than half the effective area at about the same cost) compared to AEGIS.

However, the most efficient way to build a CAT-grating spectrometer similar to IXO goals is to add it as a second instrument to a large-effective-area, long-focal-length microcalorimeter telescope, such as in the case of AXSIO (Advanced X-ray Spectroscopic Imaging Observatory),[12] a concept scaled down from IXO and developed before the RFI call, but also analyzed in the X-ray Mission Concepts Study Report (XMCSR).[11] CAT gratings offer perfect synergy with a microcalorimeter telescope, since - unlike reflection gratings - CAT gratings become transparent at higher x-ray energies where the microcalorimeter detector provides superior energy resolution,[4,10] enabling simultaneous measurements over the combined bandpass with minimal loss of effective area at higher energies. This combination provides an optimized product of collecting area times resolving power over the broadest range of energies accessible with just two instruments. The XMCSR states: “Unlike single-instrument missions, however, AXSIO also has unique complementary capabilities that are required to address some IXO (and NWNH) goals. Two examples demonstrate these synergistic efforts. The IXO science plan to address the question, “How does large scale structure evolve?” combines absorption spectroscopy using grating observations of background AGN and imaging spectroscopy of galaxy clusters. Similarly, understanding how black hole winds form and propagate requires high-resolution spectroscopy over a broad bandpass from 0.1 to 10 keV, capabilities only possible using both grating and calorimeter spectrometers.”

The XMCSR also analyzed a “calorimeter-only” notional mission concept (N-CAL) and estimated that the addition of a grating spectrometer to N-CAL would only add $\sim$ \$60 - 90M (or $\sim$ 5 - 7.5%) in cost. Furthermore, the report pointed out the well-known fact that the addition of a second instrument reduces the non-trivial risk of complete mission failure in the case of a loss of one instrument. Thus the gain of adding a CAT-XGS to an N-CAL-type telescope is tremendous, by not just extending the high-resolution spectroscopic bandpass by a whole order of magnitude into the extremely rich soft x-ray regime, but also by creating additional synergies that make the whole better than the sum of its parts, and by reducing risk - all at rather moderate additional cost.

Most of the above mission concepts assume telescope optics with an angular resolution of 10 arcsec (half-power diameter), a goal that should be achievable within the next few years. Looking further into the future, the Square Meter, Arcsecond Resolution X-ray Telescope (SMART-X)[13] concept follows the same compelling idea as AXSIO or N-CAL plus CAT-XGS, pairing large-area optics (now with 0.5 arcsec angular resolution) with a microcalorimeter at the focus and an insertable CAT grating array with an active pixel sensor readout as a second instrument.

Going back to the nearer future, a new mission is expected to start in the 2017 time frame in anticipation of the 2018 launch of the James Webb Space Telescope. Following the NWNH report this should be the Wide Field Infrared Survey Telescope. As outlined in the 2012 NASA Astrophysics Implementation Plan[14] NASA is also considering as a contingency a probe scale mission, with one of the options being an x-ray mission. It is therefore imperative that the required key technologies achieve Technology Readiness Level (TRL) 6 in the 2018-20 time frame.[15]

In the following we describe the current status of CAT grating technology development with emphasis on our progress during the last year. We also present our future plans and technology development roadmap to reach TRL6 by the end of 2018.

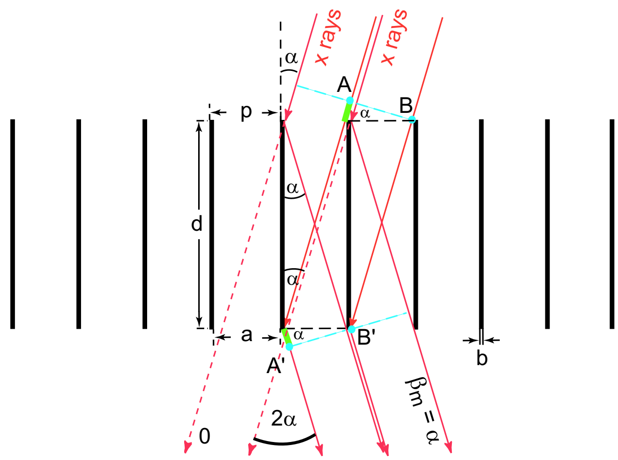

Figure 1. Schematic cross section through a CAT grating. The m^{th} diffraction order occurs at an angle β_m where the path length difference between AA' and BB' is $m\lambda$. Shown is the case where β_m coincides with the direction of specular reflection from the grating bar side walls ($\beta_m = \alpha$), i.e., blazing in the m^{th}order.

2. CAT GRATING PRINCIPLE

Critical-angle transmission (CAT) gratings are free-standing transmission gratings with ultra-high aspect-ratio grating bars. They can be described as blazed transmission gratings and combine the advantages of past-generation transmission and blazed reflection gratings.[10,16–18] The basic structure of a CAT grating is shown in Fig. 1 in cross section. X rays are incident onto the nm-smooth side walls of thin, ultra-high aspect-ratio grating bars at an angle α below the critical angle for total external reflection, θ_c (e.g. $\theta_c = 1.7°$ for 1 keV photons reflecting off a silicon surface). For optimum efficiency the grating depth $d = a/\tan\alpha$ (a being the distance between two adjacent grating bars), the grating bar thickness b should be as small as possible, and the gratings should be free-standing.

We have previously fabricated small CAT grating prototypes with periods of 574[16,17,19] and 200 nm[4,17,18,20,21] with anisotropic wet etching of lithographically patterned <110> silicon-on-insulator (SOI) wafers in potassium hydroxide (KOH) solutions. We have achieved small grating bar duty cycles ($b/p < 20\%$), unprecedented grating bar aspect ratios (d/b up to 150), and smooth side walls. X-ray tests have shown that our grating prototypes perform at the level of 50-100% of theoretical predictions for ideal CAT gratings over a broad wavelength band.[16,18]

3. LARGE-AREA CAT GRATINGS

Future space-based soft x-ray spectrographs require grating arrays to span areas on the order of 5,000 cm^2 or more. This is best achieved by tiling the area with reasonably sized grating facets that can be fabricated with standard semiconductor equipment.[4,22] Taking cost and optical design into account grating facets should be roughly between 30×30 mm^2 and 60×60 mm^2 in size. Grating facets consist of a thin machined frame that can be mounted to a large Grating Array Structure. The frame supports a thin membrane made from a <110> silicon-on-insulator (SOI) wafer. The ∼ 0.5 mm thin handle layer (back side) of the SOI wafer is machined into a high-throughput honeycomb mesh with cell sizes on the order of one to a few mm using Deep Reactive-Ion Etching (DRIE). This so-called Level 2 or L2 mesh provides the necessary stiffness and strength to the membrane. The SOI device layer (front side) is only as thick as the design depth of the CAT grating bars (∼ 4 to 6 μm). It has the grating bars and a Level 1 (L1) cross support mesh (grating with ∼ 5 to 20 μm period) simultaneously etched into it via DRIE. The buried oxide (BOX) layer that separates device and handle layer and serves as an etch stop is removed at the end, creating a free-standing grating structure with two integrated levels of support as part of the membrane (see Fig. 2).

We have fabricated CAT grating membranes that span 31×31 mm^2 with L1 and L2 supports that combined block less than 40% of the grating area.[23,24] See also Fig. 3.

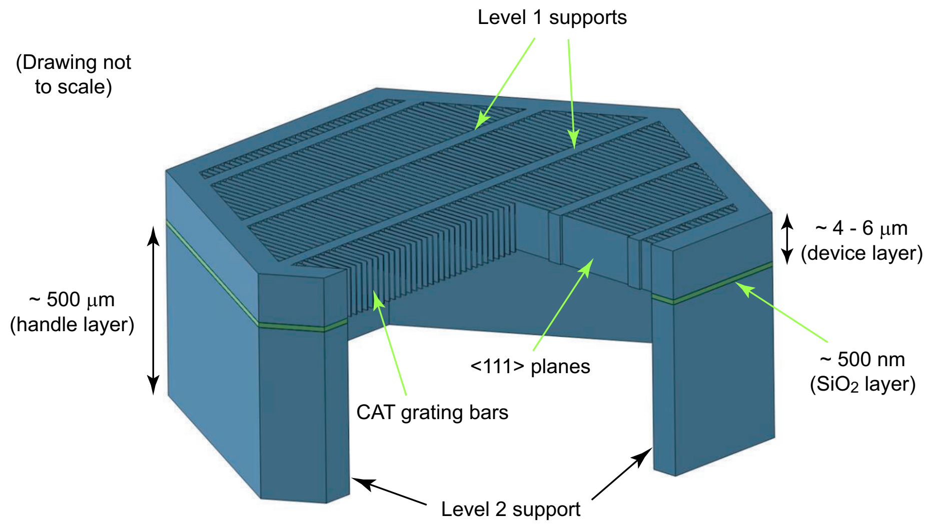

Figure 2. Schematic of a grating membrane "unit cell" (not to scale), formed by a single L2 support mesh hexagon. The L2 mesh is etched out of the SOI handle layer (back side). The device layer contains the fine-period CAT grating bars and in the perpendicular direction the coarse, low duty cycle integrated L1 support mesh. Device and handle layers are separated by the thin buried silicon oxide layer that serves as an etch stop for both front and back side etches.

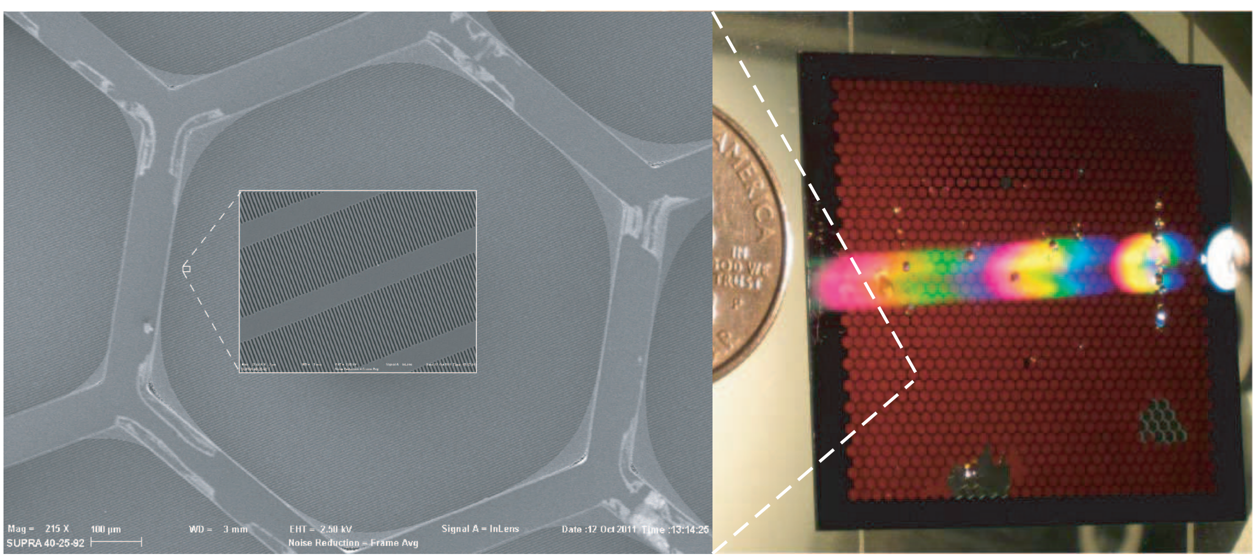

Figure 3. Large CAT grating membrane. Left: Scanning electron micrograph (SEM) of the back side of a grating membrane. The hexagon period is $\sim$ 1 mm, and the L2 mesh lines are $\sim$ 100 μm wide. The insert shows a small area of the membrane back side at larger magnification with L1 supports clearly visible. The much finer CAT grating bars are held in place by the L1 supports. Right: Grating membrane next to a U.S. quarter coin. Diffraction is due to the L1 support mesh. The hexagonal L2 mesh is visible due to back illumination. Most membrane defects were caused by intentional tearing for cross-sectional SEM studies.

4. RECENT CAT GRATING FABRICATION PROGRESS

Here we summarize our progress in CAT grating fabrication over the last year.

4.1 "Polishing" of rough sidewalls through wet etch in KOH

While DRIE can provide nearly vertical etches that allow us to minimize the size of the supports, it leaves the CAT grating bar sidewalls too rough. Over the last year we have developed a procedure that enables us to follow the device layer DRIE step with a short wet etch in KOH that reduces the sidewall roughness down to ~ 1 nm rms. For this KOH "polishing" step to work the CAT grating bars have to be aligned precisely to the silicon <111> planes that are normal to the <110> device layer surface, and the grating bar profile resulting from the DRIE step has to be close to ideal (see Bruccoleri *et al.* for more detail).[25–27]

4.2 Acquisition of a dedicated DRIE tool

Achieving the above was made more difficult due to the fact that we had to perform all of our DRIE steps remotely in a tool at the University of Michigan. At least two DRIE steps had to be performed per sample, with intermediate processing to be done at MIT. Over the last year we have prepared for the acquisition of a dedicated DRIE tool to be installed at the Space Nanotechnology Laboratory at MIT. We produced a large number of patterned and masked samples and sent them to several commercial DRIE tool vendors for front and back side etch demonstrations. After several rounds of samples and careful evaluations and analysis of the results we are now finalizing the tool purchase and hope to have the etcher installed and running before the end of the year.

4.3 Thin film stress of silicon oxide layers

Thin membranes buckle or wrinkle when subjected to compressive stress. In the case of CAT gratings this could lead to undesired variations in grating period. The thermally grown BOX layer in standard SOI wafers is under compressive stress, which can lead to fabrication problems when large areas of silicon are removed from the handle layer - as is the case at the end of the DRIE step that etches the L2 hexagons into the handle layer - and the remaining membranes (consisting of only BOX and device layer) are free to buckle. This is one of the reasons why we etch the thin device layer before the hundred times thicker handle layer, since DRIE of a buckled device layer is unproductive. Though the buckling often disappears after removal of the BOX layer with hydrofluoric acid, even temporary buckling has the potential to cause invisible structural damage to the DRIE'd CAT grating bars that may leave them vulnerable to destruction during the subsequent KOH polishing step. Also, buried oxide remains in areas that are covered by the remaining handle and device layer silicon structures (see Fig. 2).

In order to keep thin membranes flat they should be under moderate tensile stress. Ideally we would therefore like to replace the compressively strained silicon oxide layer (which is an excellent etch stop for DRIE of silicon) with an equally good etch stop layer that is under tensile stress. Over the last year we have investigated the stress and the etch selectivity of thin silicon oxide films grown from tetraethoxysilane (TEOS) precursor via dual-frequency Plasma Enhanced Chemical Vapor Deposition (PECVD). Low-frequency deposited films are porous and have tensile stress, while high-frequency grown films are dense and have compressive stress.[28] By changing the time ratio of low to high frequency deposition one can control the average oxide film stress. However, films in previous work often showed unstable stress and low etch selectivity relative to silicon. Also, if tensile films are grown too thick (critical thickness $>\sim$ 700 nm in our case) they will crack.

We were able to grow thick ($>$ 1 - 2 μm) and stable silicon oxide films with dual-frequency PECVD of TEOS with moderate tensile stress and etch selectivity comparable to thermally grown oxide. This was achieved through iterating growth (up to ~ 600 nm thickness) and annealing steps. More details will be found in Dong *et al.*[29] Fig. 4 shows a 5×4 mm^2 membrane released from such a thick oxide film on silicon without any buckling. In the future these tensile films can replace the BOX layer in SOI wafers. We expect such a modification to improve process latitude and yields for a number of CAT grating fabrication steps, to improve the mechanical stability of CAT grating membranes, and perhaps to enable alternative fabrication approaches.

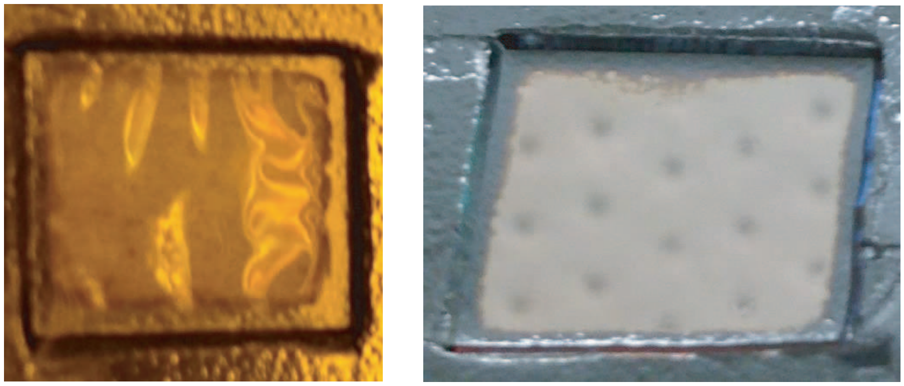

Figure 4. Left: Freestanding membrane of silicon oxide film grown thermally on silicon. Silicon was masked and etched from the back to free the oxide film. Compressive stress leads to obvious buckling. Right: Freestanding membrane of silicon oxide film grown on silicon via PECVD from TEOS precursor. Silicon was removed the same way as for the left image to form the membrane. Due to tensile stress the membrane is flat. (The "dimples" are part of a clean room wipe visible through the transparent oxide membrane.) Both films are ∼ 1 μm thick. See Dong *et al.*[29] for details.

5. FUTURE PLANS AND TECHNOLOGY DEVELOPMENT ROADMAP

As the next step in grating fabrication we need to integrate the KOH polishing step into the process flow for large-area grating fabrication.[24] Once this is achieved we will verify grating performance through diffraction efficiency measurements. We can then proceed and demonstrate CAT grating resolving power in an x-ray imaging system. For example we can use Technology Development Module optics from Zhang *et al.*[30] in the Marshall Space Flight Center Stray Light Test Facility as an imaging system. However, this facility will currently not be able to allow demonstrations of $R = 3000$ due to hardware limitations.[31] It is far from trivial to find or construct a facility that has this capability today. Alternatively, we are considering methods to measure the CAT grating period over the whole area of a grating facet with high enough precision (similar to quality control measurements on the transmission gratings for Chandra),[32] since this will be the dominant limiting factor in achieving $R = 3000$. The optical design of a CAT grating spectrometer is well understood,[4,33] and the alignment and grating flatness requirements are within present day standard technology and engineering capabilities. Nevertheless, in order to increase the TRL to 4 and 5 it will be necessary to demonstrate high-quality grating facets (membranes fabricated from SOI wafers and mounted to a frame) and to build a brass board grating array that is populated with several high-quality, large-area grating facets, and for the array to pass repeated x-ray and environmental testing. With appropriate funding, manpower and facilities access we can reach TRL5 by the end of 2016 and TRL6 by the end of 2018. More details can be found in the NASA Technology Development Roadmap for a near-term probe-class X-ray astrophysics mission.[15]

6. SUMMARY

A large-area grating spectrometer is the only choice for greatly improved high-resolution studies of astrophysical sources in the soft x-ray band feasible for launch within this decade. X-ray astronomy would gain the most from a mission that combines a microcalorimeter at the focus of a large-collecting-area telescope with a CAT grating spectrometer. The CAT grating principle has been demonstrated experimentally, and we are proceeding with the fabrication development and testing of large-area CAT gratings. Our technology development is on schedule, and with appropriate funding we can achieve TRL5 by the end of 2016 and in time for a potential probe-class x-ray astrophysics mission start in 2017.

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