

Sub-micron grating fabrication on hafnium oxide thin-film waveguides with focused ion-beam milling

Kalyani Chaganti, Ildar Salakhutdinov, Ivan Avrutsky, and Gregory W. Auner

Department of Electrical and Computer Engineering, Wayne State University, Detroit, MI-48202.
kalyani@wayne.edu

John Mansfield

Department of Space Research, University of Michigan, Ann Arbor, MI-48109.

Abstract: Uniform period sub-micron gratings have been fabricated using focused ion beam milling on hafnium oxide waveguides. Atomic force microscopy indicates that the gratings have smooth and uniform profiles. At the period of 330 nm, the largest peak-to-peak height that was achieved was 85 nm. Scattering at the grating imperfections was found to be at least two orders of magnitude weaker than the intensity of the diffracted order.

©2006 Optical Society of America

OCIS codes: (050.1950) Diffraction gratings; (130.3120) Integrated optics devices.

References and links

1. Toshiaki Suhara and Hiroshi Nishihara, "Integrated optics components and devices using periodic structures," *IEEE J. Quantum. Electron.* **22**, 845-867 (1986).
2. Don. S. Goldman, P. L. White, and N. C. Anheier "Miniaturized spectrometer employing planar waveguides and grating couplers for chemical analysis," *Appl. Opt.* **29**, 4583-4589 (1990).
3. Kenji Kintaka *et al.*, "Integrated waveguide gratings for wavelength-demultiplexing of free space waves from guided waves," *Opt. Express* **12**, 3072-3078 (2004),
<http://www.opticsexpress.org/abstract.cfm?URI=OPEX-12-14-3072> .
4. Shogo Ura, T. Kimura, T. Suhara and H. Nishihara, "An integrated-optic device using electrooptic polymer waveguide on Si Substrate for modulating focus spot intensity distribution," *IEEE Photonics Technol. Lett.* **5**, 1291-1293 (1993).
5. Kalyani Chaganti, Ivan Avrutsky and Gregory W. Auner, "Building a diffractive imaging micro spectrometer," presentation FWH36, Frontiers in Optics/Laser Science XX conference, Rochester, New York (Oct 10-14, 2004).
6. Ji Cheng and A. J. Steckl, "Focused ion beam fabricated microgratings for integrated optics applications," *IEEE J. Sel. Top. Quantum. Electron.* **8**, 1323-1330 (2002).
7. V. G. Taeed, D. Moss, B. Eggleton, D. Freeman, S. Madden, M. Samoc, B. Luther-Davis, S. Janz, and D. Xu, "Higher order mode conversion via focused ion beam milled Bragg gratings in Silicon-on-Insulator waveguides," *Opt. Express* **12**, 5274-5284 (2004),
<http://www.opticsexpress.org/abstract.cfm?URI=OPEX-12-21-5274> .
8. Robert H. Austin, Jonas O. Tegenfeldt, Han Cao, Stephen Y. Chou and Edward C. Cox, "Scanning the controls: Genomics and Nanotechnology," *IEEE Transactions on Nanotechnology* **1**, 12-18 (2002).
9. Gang Xiong, D. A. Allwood, M. D. Cooke, and R. P. Cowburn, "Magnetic nanoelements for magnetoelectronics made by focused-ion-beam milling," *Appl. Phys. Lett.* **79**, 3461-3463 (2001).
10. R. Waldhausel, B. Schnabel, P. Dannberg, E-B Kley, A. Brauer and W. Karthe, "Efficient coupling into polymer waveguides by gratings," *Appl. Opt.* **36**, 9383-9390 (1997).
11. M. A. Bader, C. Kappel, A. Selle, J. Ihlemann, M. L. Ng and P. R. Herman, "Fabrication of sub-micron gratings in ultrathin films by 157-nm laser ablation and their application as grating waveguide structures," *Proc. SPIE* **5578**, 559-567 (2004).
12. A. Katzir, A. C. Livanos, J. B. Shellan and A. Yariv, "Chirped gratings in integrated optics," *IEEE J. Quantum. Electron.* **13**, 296-304 (1977).
13. Detlef Heitmann and Carmen Ortiz, "Calculation and experimental verification of two-dimensional focusing grating couplers," *IEEE J. Quantum. Electron.* **17**, 1257-1263 (1981).

14. I. Chyr and A. J. Steckl, "GaN focused ion beam micromachining with gas-assisted etching," *J. Vac. Sci. Technol. B* **19**, 2547-2550 (2001).
15. K. Zinoviev, C. Dominguez and A. Vila, "Diffraction grating couplers milled in Si₃N₄ rib waveguides with a focused ion beam," *Opt. Express* **13**, 8618-8624 (2005), <http://www.opticsexpress.org/abstract.cfm?URI=OPEX-13-21-8618>.
16. Ning Yu and Andreas A. Polycarpou, "Use of the focused ion beam technique to produce a sharp spherical diamond indenter for sub-10nm nanoindentation measurements," *J. Vac. Sci. Technol. B* **22**, 668-672 (2004).
17. M. J. Adams, *An Introduction to Optical Waveguides* (John Wiley and Sons, New York, 1981).
18. A. V. Tishchenko, "Phenomenological representation of deep and high contrast lamellar gratings by means of the modal method," *Opt. Quantum Electron.* **37**, 309-330 (2005).

1. Introduction

Planar waveguide gratings are important building blocks in the construction of rigid and compact integrated optical devices that offer applications in many fields [1] including chemical and biological analyses. One such device that was developed for chemical analysis is a miniaturized spectrometer that employs a buried uniform period grating in tantalum pentoxide planar waveguide [2]. Some other examples are optical waveguide demultiplexer [3] and focal-spot intensity modulator [4], both of which employ focusing grating couplers (FGCs). We proposed, earlier, a hafnium oxide thin-film micro spectrometer [5] with a diffractive optic element (DOE) that performs all the three functions of collimating, dispersion and focusing. This DOE and the aforementioned FGCs are essentially two-dimensional focusing gratings on planar waveguides.

Gratings with constant periods are non-focusing gratings and they can be fabricated by various methods like conventional ruling, holographic exposure followed by wet or dry etching, focused ion-beam (FIB) milling [6-9], e-beam lithography and etching [1-3], e-beam direct writing [1], imprinting or embossing [10] and laser ablation [11]. For the fabrication of one-dimensional focusing gratings (chirped gratings with monotonically varying periodicity) or two-dimensional focusing gratings, all the above methods are technically possible. In fact, the fabrication of focusing gratings (both one-dimensional and two-dimensional) by conventional holography methods was done about three decades ago [12, 13]. Focusing grating couplers have been fabricated by e-beam lithography technique also [1, 10]. However, holographically fabricated focusing gratings pose aberration problems for usage in a wide spectral range [1, 13] which is essential for several spectrometry applications. By using FIB milling, these problems could be minimized or eliminated. FIB also facilitates ease of fabrication for unconventional micro structures.

In focused ion-beam milling, a metal ion beam, usually Ga⁺, is sharply focused on the sample along the desired pattern and the sample material is physically removed as a result of collision of high-energy ions with the sample atoms in the focused area. The amount of material removed depends on the dosage given.

Using FIB, micro and nano gratings and other structures have been fabricated on various materials including silicon [6, 7], aluminum [8], permalloy (Ni₈₀Fe₂₀) films [9], GaN [14] and recently, SiON thin film planar waveguides [15]. Diamond milling by FIB has been reported in light of fabrication of indenter for sub-10 nm nanoindentation measurements [16]. Hafnium oxide, like zirconium oxide and diamond, is a very hard and chemically stable material. Here, we report the very first fabrication of uniform period gratings on hafnium oxide waveguides by FIB.

2. Fabrication

In this work, commercially available (ThinFilm Labs) hafnium oxide thin films deposited by electron beam evaporation on 1.2 mm thick BK7 glass slides were used. We designed the thickness of the hafnium oxide films to be 293 nm. All the gratings were fabricated with an FEI 200 Nova Nanolab Dual beam Focused Ion Beam Workstation.

For hafnium oxide waveguides, surface charging occurred when the sample was subjected to the Ga^+ ion beam in the ion beam chamber. The milling side of the sample was coated with 10 nm thick chromium or carbon so as to avoid this problem. Both materials showed good adhesion to the waveguide surface and prevented the charging. Experiments were done to find the optimum settings of the FIB for the fabrication of uniform period gratings of pitch in the range 300 – 500 nm and depth in the range 50 – 100 nm. This pitch range for the gratings was chosen in order to have a good angular separation and hence, spatial separation and resolution for all the wavelengths in the targeted range (400nm -700 nm) of our integrated micro spectrometer. No particular grating profile was chosen for this experiment. It is worth noting here that in waveguide grating couplers, when high spectral and/or angular selectivity of coupling/out-coupling is required, the diffraction efficiency of the grating has to be rather low (often < 1 %) to provide large interaction length (say, > 100 μm) between the free-space beam and the guided mode. This can be easily understood using a simplified geometric-optical model: a light ray in a guiding slab has to hit the grating hundreds of times before being outcoupled. A grating with very large efficiency (approaching 100 %) would have no spectral or angular selectivity at all.

We have found in our previous experiments with hafnium oxide planar waveguide gratings that a grating depth in the range 50-100 nm provides efficient coupling of light into single mode hafnium oxide waveguides with a top silica cladding layer of 75 nm. The coupling gratings for these experiments were uniform period gratings and were fabricated on the top silica cladding layer by deep UV (244 nm) holographic exposure and dry etching [Fig. 1(a)]. Figure 1(b) below shows the light track resulting from coupling He-Ne laser light into the waveguide through the etched grating (Grating diffraction efficiency: 7 %). It also justifies our targeted depth range for gratings by FIB milling. It is of our interest to eliminate the cladding layer and fabricate gratings directly in the hafnium oxide guiding layer because hafnium oxide being chemically and environmentally stable provides very good compatibility for biological and chemical samples to be analyzed using our microspectrometer. Hence, hafnium oxide planar waveguides without any cladding layer were used for our FIB milling experiments.

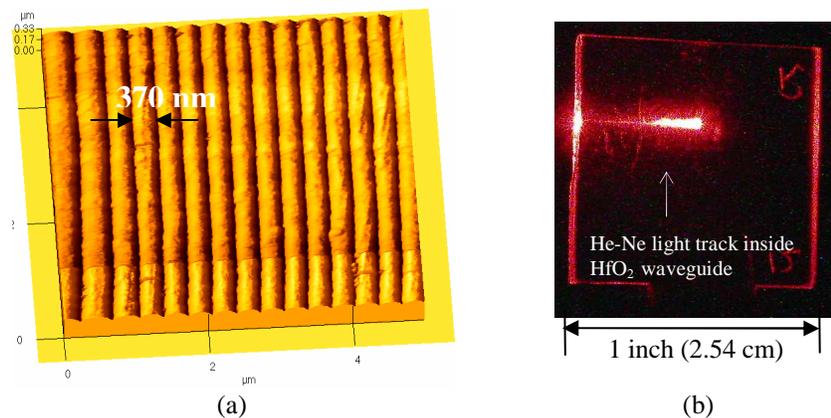


Fig. 1. (a) AFM image of a 75 nm deep uniform period grating in the top silica cladding of a hafnium oxide waveguide made by UV holographic exposure followed by dry etching (b) More than half-inch (1.27 cm) long He-Ne light track in the waveguide when coupled through the dry etched grating .

A simple program that incorporates ‘line’ command in an iterative manner was used to pattern uniform period gratings with the desired pitch, depth and length. For all the gratings, the beam overlap percentage was 50% in x and y directions and the beam dwell time was 1 μs . The voltage was kept constant at 30 KV whereas the current and programmable depth settings

were varied for different trials. Current was varied from 0.3 nA to 3 nA and the programmable depth was varied between 50 nm and 200 nm. The pitch was programmed to be either 400 nm or 500 nm. Good surface quality gratings with pitch and depth in the required range were obtained for 0.3 nA current, a ‘2000×’ magnification and a depth setting of 200 nm with ‘silicon’ as the material setting. Table 1 summarizes the settings and results for focused ion beam milling of uniform period sub-micron gratings in hafnium oxide films.

Table 1. FIB settings and milled depth results for HfO₂ films

<i>Serial No</i>	<i>Current</i>	<i>Depth setting</i>	<i>Magnification</i>	<i>Average milled depth</i>	<i>Average grating depth</i>	<i>Grating profile</i>
1 [Fig. 2(a)]	0.3 nA	200 nm	2000×	90 nm	60 nm	Smooth
2*	0.3 nA	200 nm		110 nm	85 nm	Smooth
3	0.3 nA	100 nm		60 nm	30 nm	Smooth
4	0.3 nA	50 nm		40 nm	22.5 nm	Smooth
5	1 nA	50, 100, 200 nm		50 nm, 100 nm, 220 nm	Negligible	None
6 [Fig. 2(b)]	3 nA	200 nm	1000×	370 nm	25 nm	Rough

* indicates best result. For all the above trials, the voltage setting was at 30 KV and the programmable pitch was 400 nm.

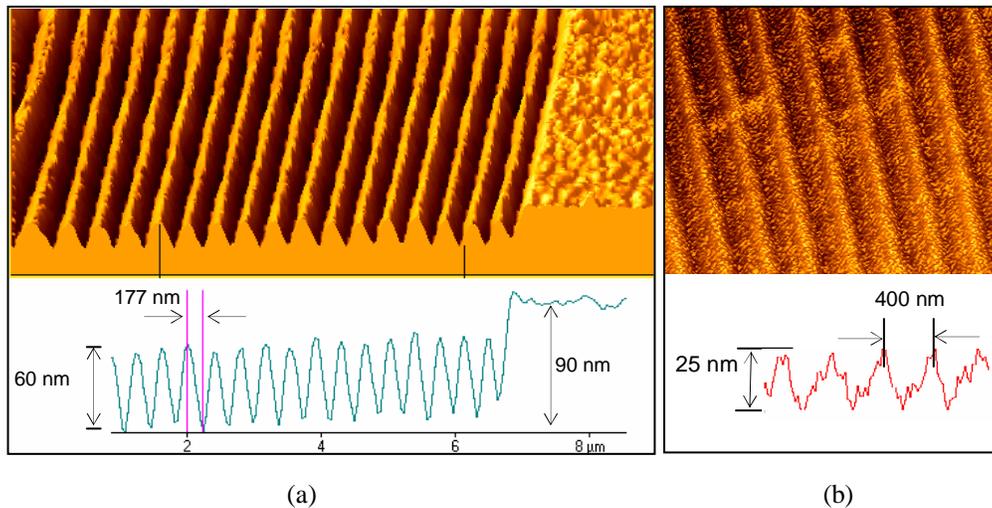


Fig. 2. (a) A typical good surface quality uniform period grating on hafnium oxide waveguide made with FIB milling, and its profile (b) AFM image of an over-milled grating fabricated using larger than optimal current (3nA), and its profile.

The deepest fabricated grating had a peak-to-peak height or in other words, grating depth of 85 nm. For all the gratings, the grating depth was lesser than the actual milled depth. The actual milled depth is a measure of how deep the sample material was milled and it can be found by taking an AFM scan near the edge of the grating. It is the perpendicular distance between the unmilled sample surface and the troughs of the grating. This lesser grating depth can be attributed to the larger extent of overlap of adjacent milled patterns which depends on the particular current setting and grating pitch. This is evident from Fig. 2(a) in which the

grating edge is seen to be at a greater height than the groove tops. The resulting grating profile was similar to the typical profile for gratings milled with FIB on silicon or Si_3N_4 [6, 9, and 15].

It has been observed that accurate focusing is very critical for the fabrication of good gratings with the required characteristics. Also, the magnification has been found to play an important role during fabrication because it represents the total area the ion beam sees at a given time and therefore, appropriate magnification setting must be done for good feature resolution for the gratings. The ion-beam was focused normally on the sample.

Consistently with results reported in [6], higher than optimal currents gave us deeper milling but shallower and rougher grating grooves for the same pitch [Fig. 2(b)]. On the other hand, a minimum amount of current is required for milling a particular grating pattern at any given magnification and hence the current values cannot be reduced below a particular value for that pattern even though lesser currents would result in lesser overlap and larger grating depths.

3. Optical characterization

For optical characterization, first, the out-coupling by the FIB milled gratings was demonstrated. A planar slab waveguide of hafnium oxide (waveguide thickness 293 nm) on which four gratings were milled and whose edges were prepared by polishing, was used for this experiment. Light from a He-Ne laser was edge-coupled to the HfO_2 waveguide with the help of a 10x microscope objective with numerical aperture $NA = 0.25$ [Fig. 3(a)]. The effective thickness [17] of this waveguide at 632.8 nm for TE polarization is 484.6 nm and the focal spot size produced by the objective at the waveguide edge is $\sim \lambda / NA \sim 2.5 \mu\text{m}$. Since the effective waveguide thickness is less than the spot size, the coupling efficiency from this edge-coupling is low but a small amount of coupling is sufficient for our out-coupling demonstration experiment.

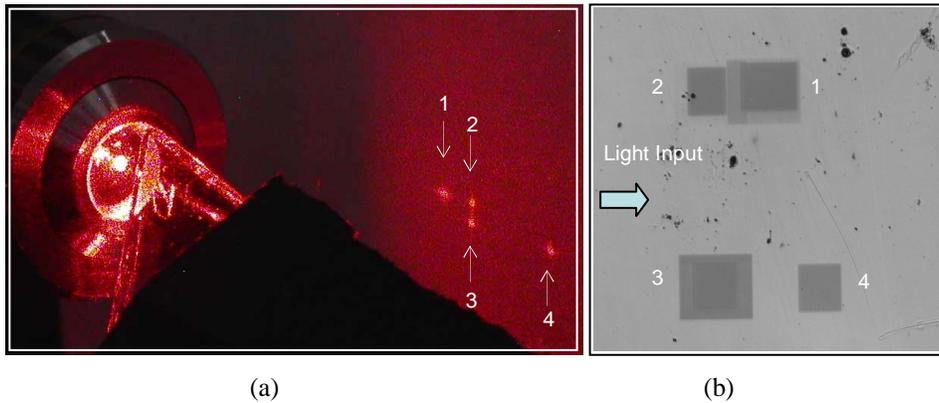


Fig. 3. (a) He-Ne laser light out-coupling from the hafnium oxide waveguide through four FIB milled gratings. (b) Optical micrograph showing the scheme of the four FIB milled gratings Grating 1: Pitch 357 nm, depth 60 nm, size $40 \mu\text{m} \times 50 \mu\text{m}$; Grating 2: Pitch 400 nm, depth 60 nm, size $40 \mu\text{m} \times 40 \mu\text{m}$; Grating 3 (the dark square area within the bigger gray square area): Pitch 400 nm, depth 60 nm, size $40 \mu\text{m} \times 40 \mu\text{m}$; Grating 4: Pitch 460 nm, depth 60 nm, size $40 \mu\text{m} \times 40 \mu\text{m}$.

A paper screen was placed about 2 cm in front of the waveguide. The out-coupled light from four FIB milled gratings was explicitly seen as four dots on the paper screen. Figure 3(b) shows the layout of the four gratings on the planar waveguide as seen in Leica DMR microscope. The grooves of the four gratings are oriented in the vertical direction. Gratings 1 and 2 are separated from gratings 3 and 4 in the vertical direction by about 100 microns on the waveguide whereas the corresponding spots on the paper screen are separated in the same

direction by about 1mm. This is because the guided light beam is not parallel but divergent. The locations of the spots on the paper screen in the horizontal direction are determined by the locations of the gratings on the waveguide and the out-coupling angles which in turn depend on the periodicities of the gratings. For this grating scheme, the relative locations of the spots on the paper screen in the horizontal direction have been primarily determined by the differences between the out-coupling angles. It is recalled here that the angle θ (measured clock-wise from the normal to the grating) of out-coupled light of wavelength λ from a waveguide grating of period Λ and waveguide effective refractive index n^* is given by:

$$\sin \theta = n^* - \frac{\lambda}{\Lambda} \quad (1)$$

The observed spots' locations on the screen are in good qualitative agreement with the locations estimated using Eq. (1).

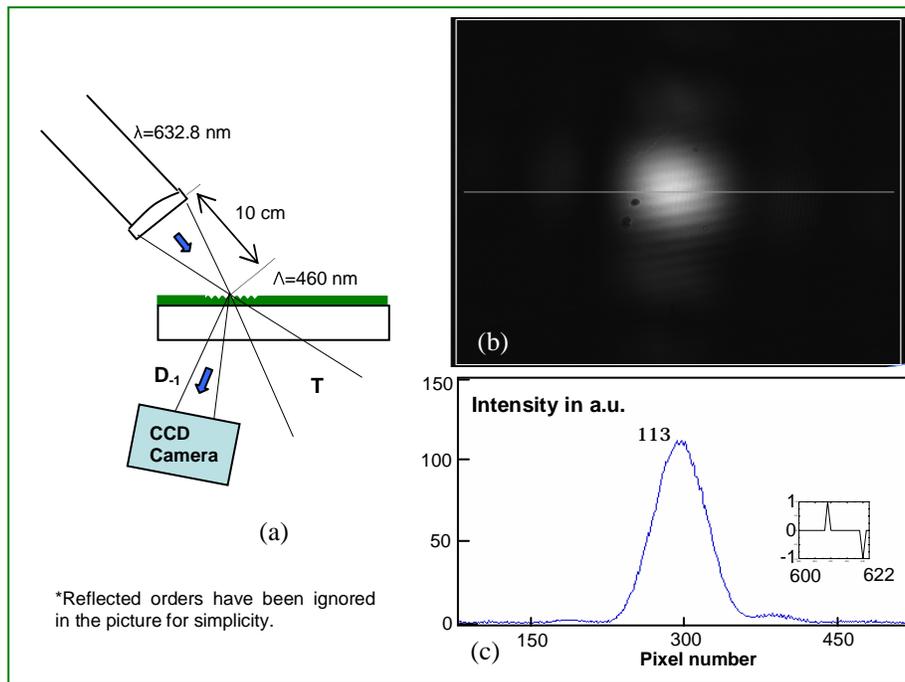


Fig. 4. (a) Experimental set-up for measuring diffraction efficiency (b) Picture of the first diffracted beam and (c) its one-dimensional intensity profile along the horizontal line shown in b. Insert shows the scatter in a section of the CCD.

The diffraction efficiency of the first diffracted order of transmission has been measured at an incident angle of 55 degrees for a typical grating whose pitch was 460 nm and depth was 60 nm (fabricated using the same settings as entry 1 in table 1 except for the programmable pitch, which was set at 500 nm). He-Ne laser beam of wavelength 632.8 nm was initially allowed to expand to about 8 mm in diameter and was then focused to a spot below 40 μm in diameter on the grating [Fig. 4(a)]. The diffraction efficiency of the '-1' transmitted order in TE polarization configuration at 55° incident angle was measured to be 0.8 %. As was mentioned earlier, for a grating coupler in a geometry shown in Fig. 3(a), the diffraction efficiency to a free-space beam is intentionally low.

Theoretical estimation of the diffraction efficiency was done using the modal method [18] assuming sinusoidal profile for the grating and it was 1.06 %. The measured efficiency is reasonably close to the estimated value. The minor difference between the estimated efficiency and the experimentally obtained value can be attributed to the grating profile which is not exactly sinusoidal and also possibly some stray scattering because of the implanted Ga⁺ ions during focused ion-beam milling.

Figure 4(b) shows the diffracted spot captured by a CCD camera. The intensity profile along a horizontal line drawn approximately through the center of the diffracted spot is shown in Fig. 4(c). The intensity profile contains the side-lobes indicating that the laser spot was partly expanded beyond the grating area. This also contributed to the reduction of the measured diffraction efficiency. The profile also contains information about both the diffracted spot and the scattering from the grating. The stray scattering from the grating at angles far from the direction of diffraction was found to be at least two orders less than the peak intensity of the diffracted beam. The weak stray scattering confirms that the grating profile achieved with optimal FIB milling is acceptable.

4. Discussion

The dry etching of hafnium oxide films hasn't been studied much so far. FIB milling might be a better candidate than e-beam lithography followed by etching for patterning in hafnium oxide films because of its proven capability of milling hard materials.

Robert Austin *et al* [8] proposed gas-assisted etching as a possible solution to the ion implanting problem and we plan to explore this in order to improve our overall integrated device performance. This would also take care of any residual carbon or chromium atoms that could have been implanted in the grating during the process of milling.

5. Conclusion

This study throws light on micro structuring in hafnium oxide thin films and is a first step towards fabrication of integrated optical components including diffractive optical elements. FIB is suitable for the milling of good quality sub-micron structures in a hard material such as hafnium oxide.

Acknowledgments

This work was supported by U.S. Army TACOM DAAE07-03-C-L140. The authors would like to thank Bill Funk for his help with Chromium metal deposition and Kai Sun and Haiping Sun for their help in focused ion-beam milling.