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Application of Plasma Chemical Etching in Control of Optical Properties of Multilayered Dielectric Gratings

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In the present research we have fabricated 2 μm period diffraction gratings in multilayered dielectric (MLD) mirror with controlled diffraction efficiencies for the 532 nm wavelength of 80 % in I_0 and 10 % in $I_{\pm 1}$ diffraction orders by plasmachemical etching (PCE) in CF_4/O_2 gas mixture plasma. Etching rate after different durations of processing was found measuring diffraction efficiency and employing atomic force microscope. Diffraction efficiency was measured with two lasers of different power and wavelength: HeNe – 632.8 nm, 30 mW, diode pumped solid state – 532 nm, 1 mW. Transmission of the multilayered dielectric structures before and after PCE processing without the mask was estimated employing a spectrometer. MLD grating fabrication conditions of controlled optical properties were defined for two different feedstock gas ratios (15 % and 20 % of O_2).

Keywords: multilayered dielectric gratings (MLD), plasmachemical etching (PCE), CF_4/O_2 plasma, diffraction efficiency.

1. INTRODUCTION

The miniaturization of micro-optics promises to revolutionize many electro-optical systems – from video cameras, video phones, compact-disk data storage to robotics vision, optical scanner and high-definition projection displays [1]. Microlenses and diffractive optical elements (DOEs), working on the principle of diffraction, require coherent and monochromatic light (usually laser beams) [1, 2]. DOEs are in general a complex pattern of microstructures, which can modulate and transform light in a predetermined way [1].

DOEs formed on dielectric mirrors show high damage threshold that enables use of the diffraction gratings providing nearly 100 % diffraction efficiency (in Littrow configuration) in the optical schemes for increasing the energy of a short pulse high-energy petawatt class laser system, while avoiding very high peak powers in the laser amplification chirped pulse amplification [3 – 5]. The multilayered dielectric (MLD) grating is usually obtained by etching a grating structure into the top layer of the high reflection (HR) mirror consisting of alternating layers of high- (HfO_2) and low-refractive index (SiO_2) materials. The grating can be etched into SiO_2 top layer, which requires special structures in order to make the MLD grating achieve high diffraction efficiency and high damage threshold [3 – 6]. K. Staliunas et. al [7] have demonstrated that periodic modulation of the refractive index in 2D photonic crystals mimicked by two multilayered dielectric mirrors with gratings shifted one respect to another by half of the grating period can substantially alter the properties of the Fabry-Perót resonator. As the transverse and longitudinal mode structure, and also the angular transmission profile of the resonator is dependent on its diffraction, then these characteristics can alter substantially due to the manipulation of diffraction.

Micro- and nano- lithography combined with dry plasma etching is widely used technology in microelectronics [8], diffractive optical elements [9, 10] and for the photonic structures fabrication [11]. Plasmachemical (PCE) and reactive ion etching (RIE) in fluorocarbon gases find many applications, e. g. for the anisotropic and selective etching of Si and SiO_2 layers. Since deposition of polymer films on the wafer surface and etching in the plasma treatment are competing processes, the etching features depend strongly on the composition of the feedstock gas and especially on the ratio of F to C [12 – 14]. Gases such as CF_4, C_2F_6, C_3F_8, CHF_3, c-C_4F_8, C_4F_6, C_5F_8 and SF_6 are employed and admixtures of H_2, Ar, CO, N_2 or O_2 are also widely used to control this ratio [12 – 14]. It is known that the radicals CF_x play an important role in etching processes by contributing to the formation of fluorocarbon polymer films and serving as precursors for etching particles like atomic fluorine [12, 13]. Additives such as O_2, Ar, and CO regulate the magnitudes of the polymerizing, etching neutral and ion fluxes incident upon the wafer, and so aid in achieving the desired selectivity and feature topology [12]. O_2 and CO addition can reduce the thickness of the polymer by oxygen atom etching. These additives ultimately control the thickness of the polymer layer by regulating fluxes for polymer formation, ion activated polymer consumption, and fluorine atom etching of the polymer. The choice of fluorocarbon gas and additives therefore determines etch rates, selectivity, and maintenance of the critical dimensions of the feature [12].

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As we have described earlier [9, 10, 15], employing plasma processing and controlling depth of the periodical structures (linear gratings) diffraction efficiency can be controlled efficiently due to additional path length in the phase diffraction gratings.

In this work we describe the technology based on micro lithography combined with PCE for the production of the MLD periodical structures with the controllable diffraction efficiency. The task of the work was to produce 2 μm period grating with the proper diffraction efficiencies, where the zero order maximum (I_0) has 80 % and first order maxima ($I_{\pm 1}$) have 10 % absolute diffraction efficiency (532 nm). The periodical structures had to be produced on the MLD mirrors used in the Fabry-Perót interferometer.

2. EXPERIMENTAL

2.1. Formation of periodical structures

Interference mirrors with the multilayer antireflection coatings, optimized for the 99 % reflection for the 532 nm wavelength and antireflection (AR) coatings for the 442 nm (angle of incidence 0°) (Altechna) formed on 2 mm thickness float glass substrate were used as substrates for the grating formation. Multilayered structure was formed from Ta_2O_5 (thickness 62 nm, $n_{532nm} = 2.167$) and SiO_2 (thickness 109 nm, $n_{532nm} = 1.457$) layers, the first layer on the top of the mirror (thickness of 180 nm) was silicon dioxide (SiO_2), which is common material used for PCE. Diffraction gratings were formed on (10×10) mm^2 substrates using standard contact-optical lithography processes and plasma chemical ion etching. Periodical structures were formed in MICROPOSIT® S1805® positive photoresist, which is optimized for G-Line (435.8 nm) exposure but it is also effective for the broad-band exposure (350 nm – 450 nm). The photoresist was deposited on the substrates employing spincoating at 4500 rpm (thickness of the layer 430 nm). The exposition (5 s – 6 s) was performed with the MA 750 contact optical lithography equipment. Residual layer of the developed resist in the grooves of the gratings was removed employing radio frequency (RF) oxygen plasma processing (RF = 13.56 MHz, $P = 0.3$ W/cm^2, $p = 133$ Pa, $t = 60$ s). The periodical structures formed in the resist were softly baked in the infrared oven ($T = 100$ °C) preparing photoresist as a mask for the plasmochemical processing. 2D structures profiles in the SiO_2 were etched in the CF_4/O_2 feedstock gas mixture (80 % : 20 % and 85 % : 15 %) RF plasma. The etching was performed using plasma-etching equipment PK-2430PD at 67 Pa pressure, total flux of gases was 300 sccm, and 0.75 W/cm^2 RF power density. After PCE processing the etching mask was removed boiling the samples in dimethylformamide (DMF).

2.2. Analytical techniques

Diffraction efficiencies of the etched structures (after boiling in DMF) were estimated employing a diffraction stand where two different wavelength lasers (HeNe – 632 nm, 30 mW (Melles Griot); diode pumped solid state (DPSS) – 532 nm, 1 mW (Apinex)) were used. Light intensities were registered with the laser power/energy display Nova II (Ophir) and photodiode head PD300-UV (Spectral range 200 nm – 1100 nm, aperture (10×10) mm^2, power ranges 10 pW – 300 mW (Ophir)). Depth of the etched structures was estimated employing AFM NanoTop-206 (MicroTestMachines) at maximum scan field area: to (20×20) μm; measurement matrix 1024×1024 points operating in a contact mode employing silicon cantilever probe with the elasticity constant 3.0 N/m. It has maximum range of measured heights: 3 μm; lateral resolution: 2 nm, vertical resolution: 0.1 nm – 0.2 nm. Pictures taken with this microscope were visualized with "Surface Xplorer" software. The optical properties (transmission/absorption) of the MLD mirrors were evaluated with an UV/VIS/NIR spectrometer (Avantes-2048), resolution 1.4 nm, with a light source – combined deuterium-halogen lamp (AvaSpec-Dhc) in the spectral range: 200 nm – 1100 nm.

3. RESULTS AND DISCUSSIONS

3.1. Etching rates and anisotropy

For the PCE we have used two different gas CF_4/O_2 ratios that enabled us to control two competing surface processes: etching and polymerization, thus to control the resultant etching rate. Due to the small period of the diffraction gratings (2 μm) the resist was softly baked in the infrared oven as described in 2.1 (when the standard baking temperatures are slightly higher: 115 °C – 120 °C [16]) trying to avoid changes in the morphology of the resist. Increase in oxygen concentration (from 15 %, to 20 %, respectively decrease of CF_4 from 85 % to 80 %) reduced the polymer thickness sufficiently to clear the feature [12] that resulted in the higher etching rate of silicon dioxide, that was evaluated optically.

3.2. Optical investigation

Periodical structures etched in SiO_2 (the first layer of the multilayered interference film formed on the float glass substrates) behave as phase diffraction gratings therefore the depth of the periodical structures strongly influences the diffraction spectrum. As the gratings were formed on multilayered AR structures, one has to consider that the zero order diffraction (I_0 specular reflection) is strongly affected by the optical properties of the AR coating. Optical properties of the multilayered AR coating before and after etching (without mask) in CF_4 plasma were evaluated with the optical spectrophotometer. As expected, no changes at 532 nm in the transmission spectra were observed, because antireflection coating for the 442 nm wavelength was above the one for the 532 nm. As one can see, in transmission spectra (see Fig. 1) PCE processing has affected mostly the 350 nm – 450 nm and 600 nm – 1000 nm AR regions.

Diffraction efficiencies measured with two different wavelength lasers after different duration of PCE treatment (after removal of the etching mask) are presented in Fig. 2. HeNe laser for the optical measurements was used due to higher power and higher diffraction efficiency signal/noise

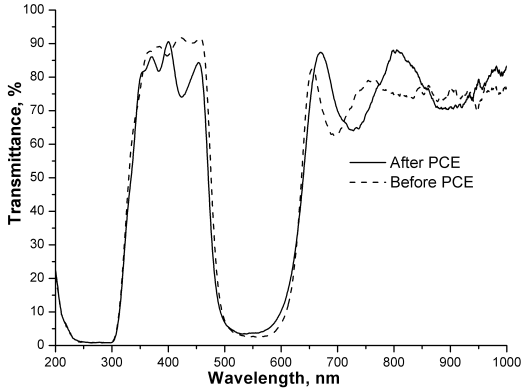

Fig. 1. Optical transmission spectra of the dielectric mirror with the top SiO_2 layer before and after chemical processing in the CF_4 plasma (CF_4 / O_2 – 85 % / 15 %, t = 120 s)

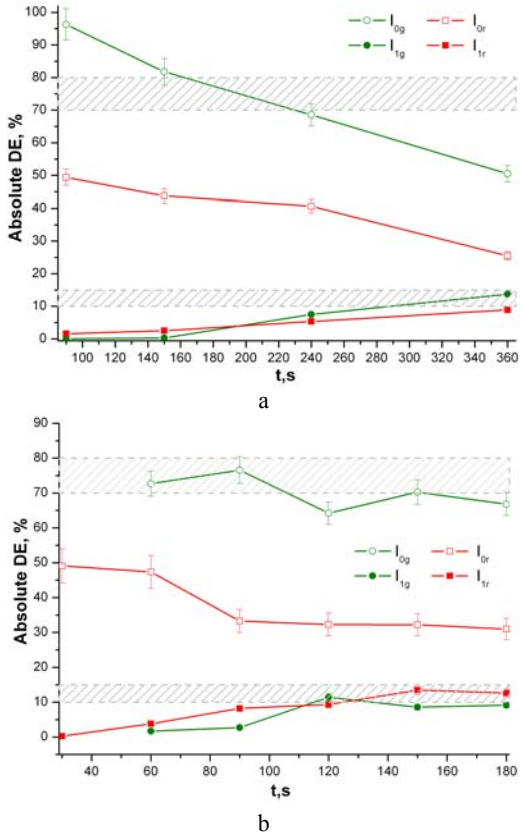

Fig. 2. Diffraction efficiency dependence on the duration of PCE treatment in CF_4 / O_2 plasma: a) CF_4 / O_2 – 85 % / 15 %, t = 90 s – 360 s, b) CF_4 / O_2 – 80 % / 20 %, t = 30 s – 180 s. "I_1" and "I_0" stands for the diffraction orders (average of $I_{\pm1}$ and I_0 respectively) and "g", "r" indicates the used wavelength (532 nm and 632.8 nm respectively). Dashed region indicates limits of the necessary diffraction efficiency

ratio for the short PCE processing durations. According to the transmittance spectra (Fig. 1) the 532 nm wavelength is strongly reflected (~85 %, if we consider that the absorption is minimal) and 632.8 nm wavelength is partially transmitted (~50 %). Therefore the zero order diffraction for the 532 nm wavelength shows higher efficiencies as compared with the 632.8 nm (see Fig. 2, a). Linear growth of the first order (I_1) diffraction efficiency versus PCE processing duration is related to the increasing depth of the etched trenches, that was confirmed by the AFM measurements, thus the increased phase difference [9, 10, 15].

3.3. AFM investigation

Depth of the periodical structures after different duration of PCE treatment was estimated employing the AFM. The AFM images (see Fig. 3, a – d) have shown that the profile formed in the photoresist with conventional contact optical lithography was transferred to the thermal SiO_2 and that the etched depth versus PCE treatment duration is close to the logarithmic function (see Fig. 4).

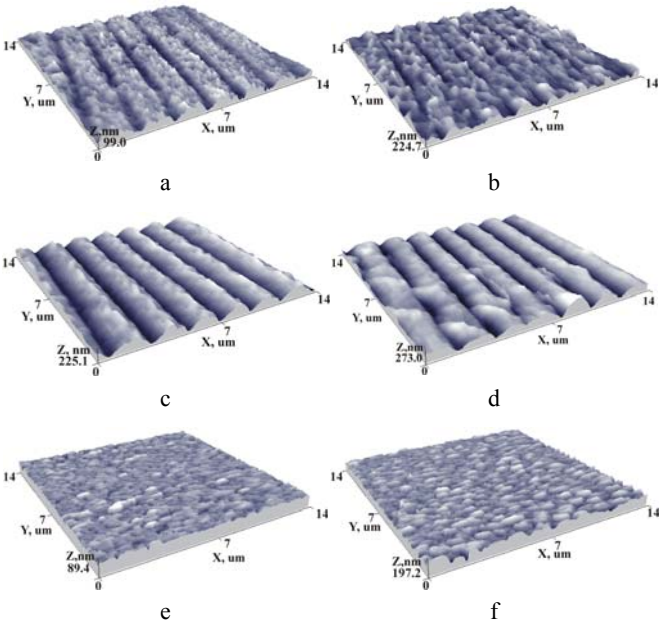

Fig. 3. AFM images of 2 μm period gratings plasmochemicaly etched in SiO_2 after different duration of etching: a) 30 s, b) 90 s, c) 150 s and d) 180 s. Substrate morphology (top layer – SiO_2 of the AR coating) before (e) and after (f) etching in CF_4 / O_2 plasma (80 % / 20 %, t = 120 s)

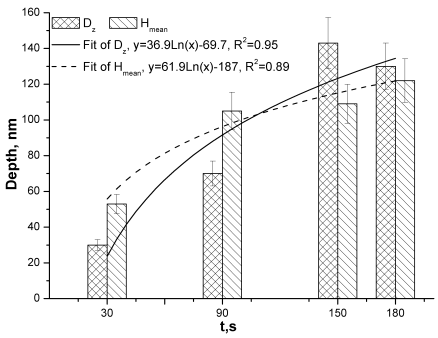

Fig. 4. Depth of the diffraction gratings (period – 2 μm), etched in SiO_2, found with AFM, dependence on the duration of PCE treatment (CF_4 / O_2 – 80 % / 20 %). "H_{mean}" indicates mean value of the heights, "D_z" indicates height difference between top and bottom of the pits of the grating structure

These dependencies can be extrapolated with: $H_{mean} = 48.5\ln(t) + 58.7$ ($R^2 = 0.921$), $D_Z = 82.54\ln(t) + 27.67$ ($R^2 = 0.883$). Roughness that can be seen on the etched gratings (see Fig. 3, a – d) is similar to the morphology of the initial and etched SiO_2 film without the photoresist mask (see Fig. 3, e – f). One can see that the surface before

treatment has roughness of 5.9 nm which increases to 16.6 nm after 2 minutes of processing. No influence of the PCE processing for the final microstructure roughness was found (see Fig. 3, a) as compared with the one of the etched flat surface (see Fig. 3, f).

4. CONCLUSIONS

We have demonstrated that employing microlithography and plasma chemical etching it is possible to transfer 2 μm period gratings formed in softly baked photoresist to the SiO_2 top layer of the MLD structure using CF_4 / O_2 plasma processing.

Optically controlling (measuring diffraction efficiency and transmission spectrum) the samples after different PCE treatment durations it is possible to optimize technological parameters (etching rates with different gas ratios) for the fabrication of the MLD diffraction gratings providing the appropriate diffraction efficiencies in the first and zero orders.

We have demonstrated that using less oxygen (CF_4 / O_2 – 85 % / 15 %) in the plasma gas mixture one can make the etching rates slower, therefore more sensitive control of the diffraction efficiencies of the possible MLD diffraction gratings.

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