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Optical Evaluation of Geometrical Parameters of Micro-Relief Structures

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In the present research we have fabricated and investigated two kinds of surface relief gratings (phase diffraction gratings), formed with standard photolithography combined with the plasma-etching and reactive ion etching (RIE). Diffraction efficiency of diffraction gratings (originally produced in silicon and quartz glass substrates) was measured experimentally and estimated using linear dimensions of gratings defined by atomic force microscopy (AFM) and scanning electron microscopy (SEM). The main experimental results were compared with the computer simulations where the standard software ("PCGrate 2000MLT" and "GSolver") were employed to calculate diffraction efficiency of different period of diffraction gratings for the different wavelengths of visible light. Comparing two evaluation methods: direct (scanning probe microscopy) and indirect (diffraction efficiency) we have demonstrated feasibility of optical methods in control of geometrical dimensions of gratings at the microscopic range. These methods enabled us to control and produce diffraction gratings for the different optical applications, like dispersive elements in spectroscopes, beam splitters or micro-fluidic devices.
Keywords: diffraction gratings, diffraction efficiency, micro-fluidic device, beam splitter.

1. INTRODUCTION

Interest in diffractive optical elements (DOEs) [1] for optical interconnects [2], optical imaging, optical data storage [3], laser machining and optical sensors has grown rapidly in recent years. The elements showing high diffraction efficiencies have good optical qualities for practical use. Various methods and materials are used to produce diffraction gratings for DOEs. Two-dimensional or three-dimensional periodic structures of micrometer period are widely used in different optical devices employing diffraction properties of these structures. Periodic microreliefs could be used as dispersive elements, beam splitters [4] or micro-fluidic devices [5, 6].

For example transmission phase diffraction gratings [7] working as beam splitters can be used in optical systems for dynamic holography experiments [8, 9] or in interference immersion lithography. Long deep groves (phase diffraction grating) can be used as elements in micro-fluidic devices. The microscale synthesis, processing, and analysis of chemical and biological samples require manipulation of microscopic volumes of liquids, which can be done with chips with micro-channels and microreactors.

For characterization of geometrical parameters of microrelief structures usually various construction microscopes are used, mostly scanning electron and probe microscopes [10]. These direct methods are expensive, sometimes destructive and hardly can be employed for in-situ analysis. Therefore indirect optical interference or diffraction methods are used widely [11].

From this point of view optical methods are very flexible and efficient in control where dimension of periodic structures in micrometer range. In [12] it was demonstrated feasibility of optical control of relief variations during different technological steps of producing diffractive elements.

In the present work we present experimental and simulation results of deep periodic structures the depth of which can be few micrometers. These deep periodic structures in silicon and fused quartz found two applications: in beam splitter and micro-fluidic devices. Two tasks were solved while producing and characterization of these structures: I – laser beam splitter – production and calculation of diffraction gratings of high diffraction efficiency with concentration of energy in first order diffraction maxima; II – evaluation of geometry of periodic grooves in silicon (micro-fluidic device).

2. EXPERIMENTAL

Diffraction gratings that were supposed to be used as beam splitters, were formed in 10 mm × 10 mm quartz "MB Whitaker & Ass – QZ" substrates [13] using standard contact-optical lithography processes and plasma chemical ion etching. 2D structures profile in quartz substrate was formed at 0.5 Torr pressure in the CF_4 / O_2 (90 % : 10 %) gas mixture, which float was 300 cm^3/min (at 1 W/cm^2 RF power density), plasma, using plasma-etching equipment PK-2430PD. The depth of diffraction gratings – 0.7 μm was obtained after 40 minutes of processing.

Micro-fluidic devices were formed on 20 mm × 20 mm silicon plates with 10 channel systems etched into it. Silicon plates 20 mm × 20 mm surfaces were chemical cleaned in boiling dimethylformamide and oxygen plasma processed (RF = 13.56 MHz, P = 0.3 W/cm^2, time 5 s – 60 s). Micro-fluidic devices were produced in <111> crystalline silicon using standard contact-optical lithography processes and reactive ion etching. 2D structures profile in Si(111) substrate was formed by RIE in the SF_6 gas [14],

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which float was 2 cm^3/min (at pressure 0.04 Tor, and 0.25 W/cm^2 RF power density), plasma, using plasma-etching equipment PK-2420RIE. The etching process lasted 7 min 20 sec.

Diffraction spectra of both beam splitters and system of channels of the micro-fluidic device were registered with the diffraction stand (He-Ne laser, $\lambda = 632.8$ nm, diffraction spectra were registered by a photodiode [15] "BPW32"). The following analytical methods were used to control microrelief: optical microscopy, atomic force microscope NANOTOP-206, scanning electron microscope JEOL SM-IC 25S.

3. SIMULATION RESULTS OF DIFFRACTION EFFICIENCY

In the present research we have simulated, fabricated and investigated surface relief diffraction gratings (phase diffraction gratings), formed with standard photolithography combined with the plasma chemical ion etching (in quartz) and reactive ion etching (in silicon).

3.1. Transmission diffraction grating (beam splitter)

Optimal diffraction spectra of a phase diffraction grating for beam splitter correspond to the case, when the zero order diffraction maximum is minimal and first order diffraction maxima are maximal. The depth of diffraction gratings for the beam splitter was selected from the modeling results done with "GSolver" software [16, 17]. The period, and the diffraction grating shape are defined by the user mask, way of etching - so in our model just the depth of rectangular diffraction grating was changed. Modeling results of the zero and first order diffraction maxima diffraction efficiencies versus depth of the phase diffraction grating are plotted in Fig. 1. One can see that absolute diffraction efficiency versus depth for both maxima varies in the same way for all calculated periods (5, 10, 15 µm).

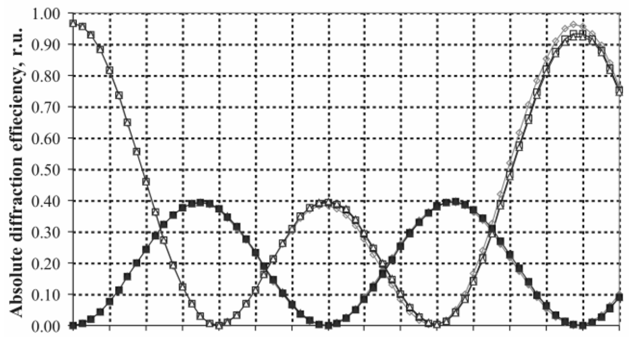


Fig. 1. Modeling results (done with "GSolver") of absolute diffraction efficiency dependence of zero (0T) and first (1T) order diffraction maxima on the depth of phase transmission diffraction grating in quartz (with period 5 µm, 10 µm, 15 µm) when the grating is illuminated perpendicular to the surface with 632.8 nm wavelength

Modeling results (Fig. 1) show that diffraction efficiency dependence on depth is a periodical function. One can understand that this phenomenon occurs due to the phase shift (additional path length difference Δ) in the phase transmission diffraction gratings. Using these results it is possible to calculate zero order diffraction maxima conditions (corresponding diffraction grating depth). To explain qualitatively this phenomenon we proposed a simple geometrical model (Fig. 2) that allows understanding and calculating optical path length difference in the grating. The Δ depends on the geometry of grating (d_1, d_{21}, d_{22}) and refraction index of the grating material (n_2):

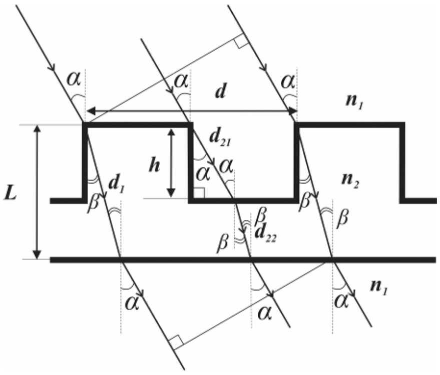


Fig. 2. Geometrical model explaining optical path length difference in rectangular phase transmission diffraction grating

$$\Delta = d_1 \cdot n_2 - (d_{21} \cdot n_1 + d_{22} \cdot n_2) . \quad (1)$$

This dependence can be easily expressed in terms of depth of the groove (h) and angle of incidence (α):

$$\Delta = hn_2\left(\sqrt{{n_2}^2 - \sin^2\alpha \cdot {n_1}^2} - \frac{n_1}{\cos\alpha}\right). \quad (2)$$

One can understand that constructive interference condition:

$$\Delta = m\lambda; m = 0, 1, 2, ... \quad (3)$$

will describe depth of diffraction grating (for fixed angle of incidence) where maximum value for the zero maximum will take place. Changing the angle of incidence (α) will result in the change of Δ, and, as result, change of position of maximum versus depth of the grooves. These dependencies (integer number of wavelengths versus depth of the grating) are summarized in Fig. 3.

The cross of the horizontal line corresponding $\Delta = 1\lambda$ or $\Delta = 2\lambda$ on Fig. 3 allows to predict depth of the grating corresponding to the maximum intensity of zero maxima (i.e. in this case they correspond to h = 1400 nm and 2750 nm). These evaluations are in good correlation with the simulation results done with "GSolver" (Fig. 1). One can understand that grating with the different depth then discussed ones will produce maxima at different angle of incidence.

In similar way, thicknesses of diffraction grating providing maximum of the zero maximum can be easily calculated putting $\Delta = (2m+1)\lambda / 2; m = 0, 1, 2, ...$. This condition defines depth of diffraction grating corresponding to the minimum of the zero order maximum (or maximum of the first order maximum). This consideration defines optimal depth of the diffraction grating in quartz, working as a beam splitter (zero order maximum is equal to zero and first order maxima have maximal value), that in our case should be equal to 0.7 µm.

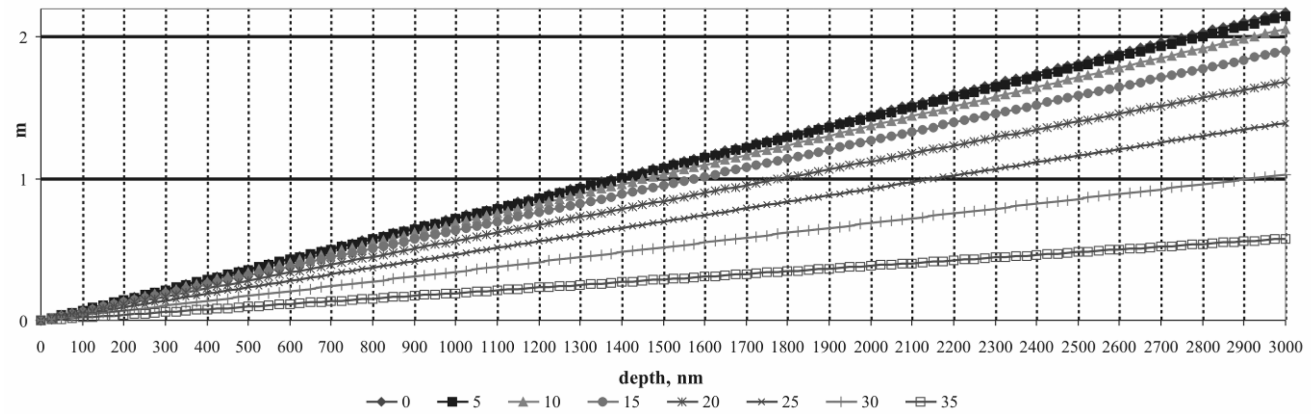


Fig. 3. Number of integer wavelengths in optical path length difference distance vs. depth of the rectangular phase transmission diffraction gratings for different angles of incidence (0°, 5°, 10°, 15°, 20°, 25°, 30°, 35°) (this dependence can be applied for any period of grating)

3.2. Reflection diffraction grating (system of channels)

"PCGrate" software [18, 19] was applied to calculate diffraction efficiency of the reflecting diffraction grating in silicon. Originally this system of deep grooves presents a fragment of channels of the micro-fluidic device. In this case the simulations were done to evaluate diffraction efficiency of the system versus depth of the grooves and angle of incidence of light. 12 μm period of the device resulted in 37 maxima. As an example, calculation results of zero order maximum versus depth of the grooves for different angles of incidence (0°, 5°, 10°, 15°, 20°, 25°) are given in Fig. 4.

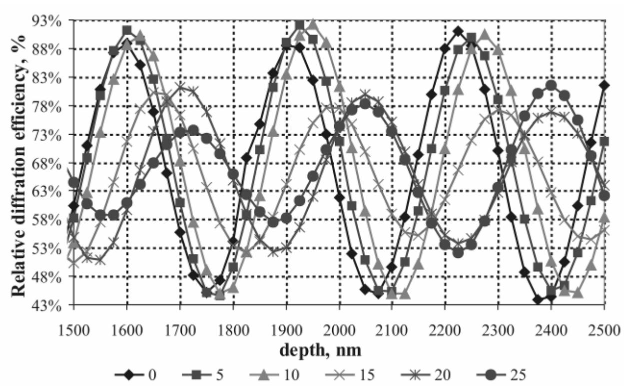


Fig. 4. Dependence of the zero order diffraction maxima on phase diffraction grating depth and irradiation angle (0°, 5°, 10°, 15°^{0}, 20°, 25°) modeling results done with "PCGrate")

According to the simulation results, diffraction efficiency dependence versus depth is a periodical function. This phenomenon can be easily understood in terms of optical path length difference produced in a reflection diffraction grating. Similarly as it was demonstrated previously, additional optical path length difference distance Δ in a phase diffraction grating in this case could be calculated using a geometrical model (Fig. 5). This optical path depends on irradiation angle *α*, material refraction index (n_2) and phase diffraction grating depth (h):

$$\Delta = n_1\left(d_1 + d_2\right); \quad (4)$$

$$\Delta = \frac{n_1 h}{\cos\alpha}(1 + \sin(90 - 2\alpha)) \; . \quad (5)$$

As it was demonstrated previously using eq. (3) i.e. dividing additional optical distance Δ versus wavelength of the visible light used for measuring, one can find zero order diffraction maxima conditions for phase reflection diffraction gratings (Fig. 6).

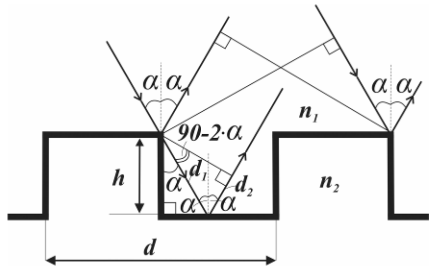


Fig. 5. Geometrical model explaining optical path length difference in rectangular phase reflection diffraction grating

4. RESULTS AND DISCUSSIONS

Validation of the simulation results was performed on the quartz beam splitter (transmission diffraction grating) and systems of channels of the micro-fluidic device (reflecting diffraction grating). AFM and SEM views of the fabricated beam splitter and micro-fluidic devices are demonstrated in Fig. 7. The depth of a beam splitter was measured with AFM (Fig. 7, a) and it is approximately 0.7 μm. To measure the depth of micro-fluidic device with AFM is a challenging task and this method can be used for the shallow profiles only (Fig. 7, b). According to the SEM measurements, the final depth of microchannels was 2000 nm and this value was used in further diffraction spectra calculations.

Comparing "GSolver" modelling results – diffraction efficiency values for 0.7 μm depth transmission diffraction grating different period (5, 10, 15 μm) with the experimentally measured values (Fig. 8), one can see that absolute values of diffraction efficiency of different diffraction maxima are in correspondence (at least qualitatively) with the predicted ones by simulation. This fact demonstrates that it is possible to control diffraction spectra changing the depth of phase diffraction grating,

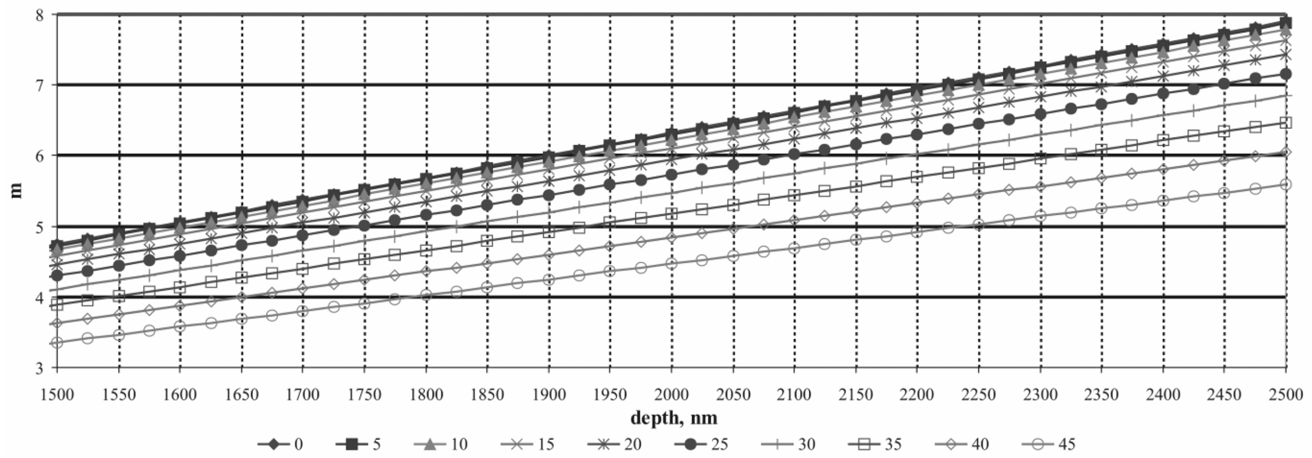
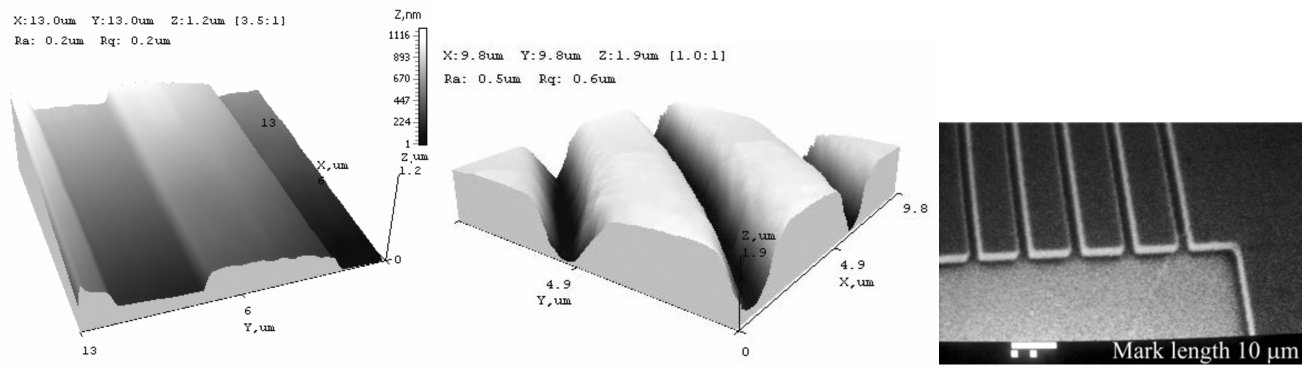


Fig. 6. Number of integer wavelengths in optical path length difference vs. depth of the rectangular phase reflection diffraction gratings produced in silicon (calculated for angles of incidence 0°, 5°, 10°, 15°, 20°, 25°, 30°, 35°, 40°, 45°) (this dependence can be applied for any period of grating)



a b c

Fig. 7. AFM and SEM views of produced periodic structures: a – AFM view of phase transmission diffraction grating in quartz – beam splitter, period 10 µm, b – AFM view of channels of micro-fluidic device in silicon after 7.3 min of RIE processing c – SEM view of channels of micro-fluidic device (the reservoir connecting the channel system is shown in the lower part)

and these changes can be predicted by our proposed simple geometrical model. This particular diffraction grating could be used as an efficient beam splitter in optical schemes.

The modelling results of reflection diffraction grating were used as well for depth evaluation of deep periodicstructures – micro-fluidic device in silicon. The main experimental results, angular dependence of diffraction spectra, were compared with the computer simulations where the standard software ("PCGrate") was employed to calculate diffraction efficiency of different depth of diffraction gratings for various illumination angles of visible light (dependencies like presented in Fig. 4).

We have calculated coefficients of different depth (c_{depth_i}) of simulated diffraction gratings, for different irradiation angles and compared them with the corresponding experimental values of diffraction efficiency (6). To find the depth of the grooves these coefficients of different irradiation angles were averaged and average value was calculated according to (a_{depth}) (7). The curve of the dependence of coefficients average on depth was plotted (Fig. 9).

$$c_{\text{depth}_i} = \frac{1}{n}\sum_{i=1}^{n}\left(DE_{\text{theoretical}_i} - DE_{\text{eksperimenatal}_i}\right)^2 ; \tag{6}$$

$$a_{\text{depth}} = \frac{1}{n}\sum_{i=1}^{n} c_{\text{depth}_i} . \tag{7}$$

One can see that $c_{\text{depth}_i} = f(h)$ is a periodical function, i.e. direct comparison of simulation results of diffraction

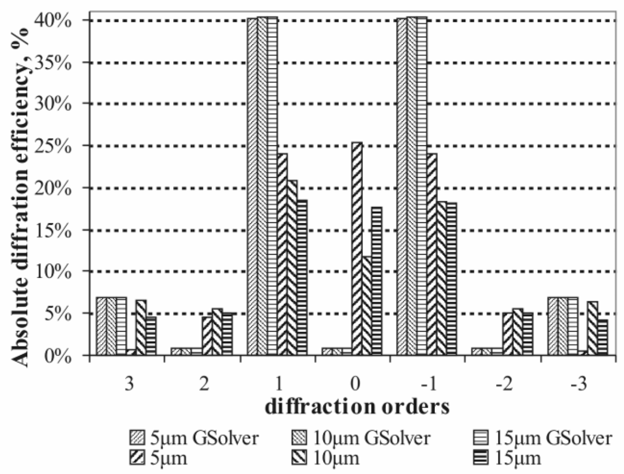


Fig. 8. Comparison of simulated (5 µm GSolver, 10 µm Gsolver, 15 µm GSolver) and experimental (5 µm, 10 µm, 15 µm) results for 0.7 µm depth transmission diffraction grating in fused quartz for λ = 632.8 nm wavelength

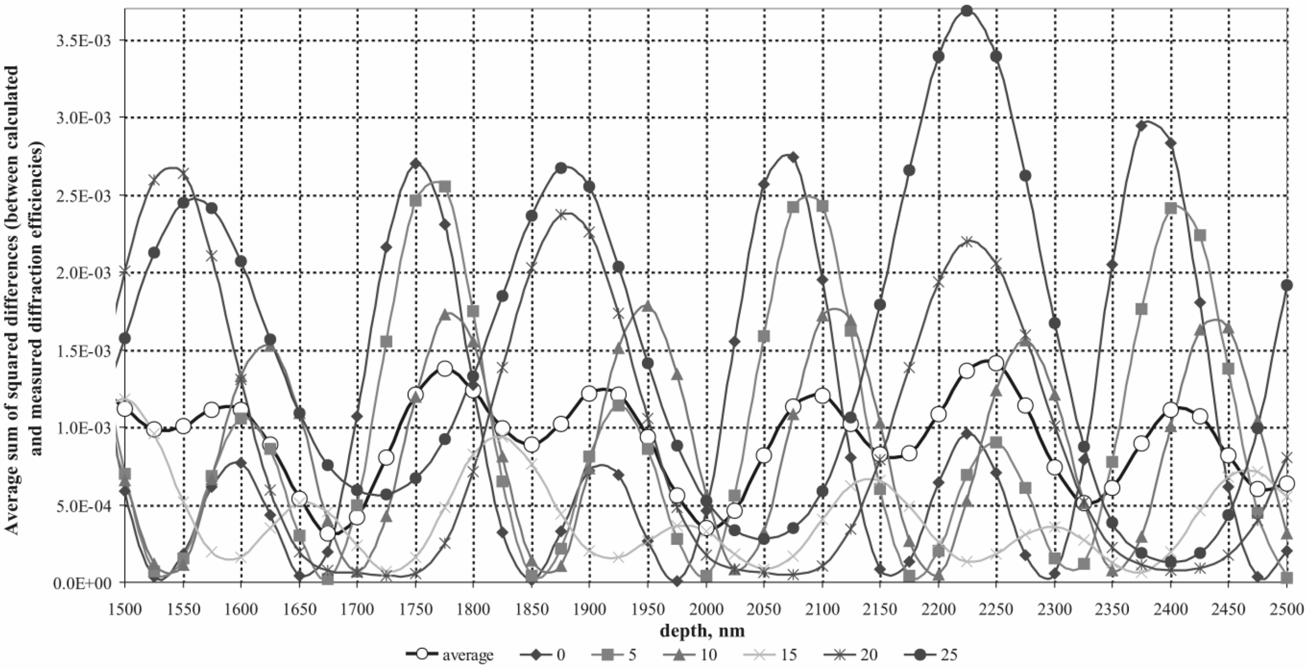


Fig. 9. Depth evaluation method where average sum of squared differences between simulated and experimentally measured diffraction efficiencies (angles of incidence 0°, 5°, 10°, 15°, 20°, 25°) are compared. "–o– average" corresponds to the results calculated according to (7)

efficiency and experimental diffraction efficiency enables to define depth of the grooves with some period only. Therefore the approximate depth should be evaluated with an alternative method: SEM, AFM or profilometer – in our case SEM was used. Comparing experimental 37 diffraction order maxima and theoretical diffraction efficiencies, the depth of the channels defined as a minimum of $a_{\text{depth}} = f(h)$ in the vicinity of 2000 nm was found to be equal to 2010 nm (Fig. 9).

CONCLUSIONS

1. Comparing two evaluation methods: direct (SEM, AFM) and indirect (diffraction efficiency) we have demonstrated feasibility of optical methods in control of geometrical dimensions of gratings at the microscopic range.

2. The modeling results for reflection and transmission diffraction gratings show that diffraction efficiency dependence versus depth is a periodical function that could be explained in terms of extra phase shift (optical path length difference).

3. Approximate depth should be evaluated with alternative method: SEM, AFM or profilometer to define the exact value of the depth (in our case the depth was measured with SEM). The evaluation of micro relief estimating diffraction spectra dependence of incidence angle could be more accurate if more than one wavelength would be employed.

4. Changing the technological parameters (depth of the diffraction grating in quartz) and using the modeling results we have achieved 40 % absolute diffraction efficiency in the firs order diffraction maxima that can be used as an efficient beam splitter for visible light of 632.8 nm wavelength.

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