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Particularity of diffraction optical elements with pattern periodicity less than 1 μm produced by ion etching*

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Particularity of ion-beam application and reactive ion-beam etching as well as holographic exposure for microstructures of high resolution, which are used as optical elements in such fields as spectral equipment, integrated and adaptive optics in wavefront reversing systems, recording and keeping of information systems, etc., are examined for the case of holographic diffraction gratings with symmetric and asymmetric profile of the pattern obtained in the layer of photosensitive composition and on the surface of optical glass. It has been shown that the main factors which determine both the symmetrical form of the profile and the blaze angle are the angular dependence of the etch rate of photoresist on the angle of ion incidence and parameters of the initial sinusoidal profile grating, i.e., relation d/h, where d is the grating period and h is the relief depth. Holographic diffraction gratings were obtained with the number of patterns of 1200–2400 lines/mm and blaze angle of 9–30°. The dependence of parameters of the obtained microstructure profile on the conditions of ion-beam and reactive ion-beam etching is investigated.

1. Introduction

In recent times, microstructures of high resolution have become the most important part of diffraction optics due to their widespread application, which plays a great role in the development of different fields of science and technology. Apart from traditional use, as dispersive elements in spectral equipment, similar microstructures are used in holographic integrated optics, quantum electronics, as well as during registration and storing of information, etc.

Diffraction structures of asymmetric profile diffraction are of main interest. The present methods of grating manufacture are difficult and offer no reliable technology to obtain microstructure of an exact profile of the pattern and the wanted energetic parameters.

As a rule, reflection "blaze" gratings [1] are fabricated by a mechanical method, that is, direct cutting of the surface relief into thin layers. However, the main difficulty of the mechanical method is the manufacturing of diffraction gratings with the number of groves per length unit as high as 1200, 2400 and 3600 lines/mm with light requirements as to the exact localization depth of the pattern elements, the

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violation of which leads to false diffraction orders and unequally dispersed light on the surface.

One of the widespread methods is the technology of holographic recording of periodical structures on the photoresistive layers. But, as a rule, these gratings have a symmetric profile of the pattern [2].

For creation of an asymmetric profile of the patern, the method of holographic exposure of the thick layer of photoemulsion in optical field of standing wave and the method based on Fourier synthesis are used [3]. However, these methods are difficult to apply in controlling the profile of the structures and obtaining the necessary blaze angles and diffraction efficiency.

As the most universal method with an exact control of the microstructure manufacture of high resolution, the method can be considered which combines the process of ion-beam or refractive ion-beam etching and holographic exposure of photosensitive layer for forming of the protective relief masks. This method permits manufacturing microstructures with either a symmetric profile on optical substrate (glass, semiconductors, crystals) and or an asymmetric profile, both in the photosensitive layer and on the surface of optical substrates [4]–[6].

2. Experiment

Organic relief masks were obtained by subsequently casting of photosensitive layers (composition on the basis of the novolac resine) on the optical substrate by means of centrifugation exposing by holographic method and developing by chemical method in a special solvent.

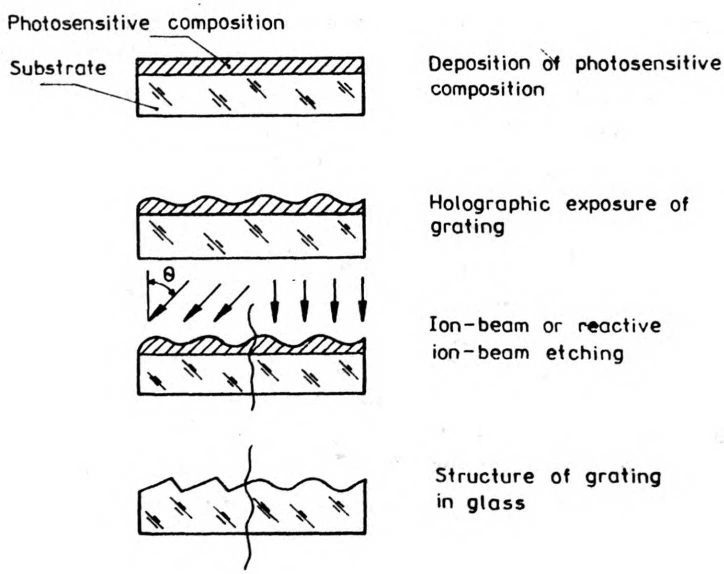


Fig. 1. Technological scheme of DOE manufacturing

Ion-beam etching and reactive ion-beam etching of the samples were carried out in vacuum system with autonomous source of ions with cold cathode which permits carrying out ion processing both in inert atmosphere and in atmosphere of active gases. The equipment was supplied with sample planetary rotating system which allows us to conduct the etching under different angles of ion incidence upon the surface.

The investigation both of the surface topology and the structure profile of holographic gratings was carried out with the use of electron microscope (EM-14, USSR). Figure 1 presents the scheme of the manufacture process of holographic gratings in glass and transformation of the grating profile, holographically recorded onto photosensitive layer.

3. Results and discussion

It is well known that during the ion bombardment of the solids, some topographic changes of their surface are developed. The change of surface topography is dependent on the differences of the etching rate as related to different angles of ion incidence. The character of the surface evolution is conditioned by the dependence of the sputtering coefficient versus the ion angle of incidence.

In this paper, the Carter theory for a simplified case is used for the analysis of evolution of the holographic gratings profile and the analysis of conditions of microstructure manufacture of needed profile and topology in optical materials.

We assume that the solid surface is uniformly bombarded by an ion bunch with constant flow density and neglect secondary effects such as local differences of flow density, reverse precipitation of dispersed atoms, migration of atoms onto the surface. In the Carter's work, the characteristic equation describing the trajectory of points on the real surface is expressed in terms of the etch rate as follows:

$$v = (v_x^2 + v_y^2)^{1/2} = \left[\left(\frac{\Phi}{N}\frac{dS(\vartheta)}{d\vartheta}\cos^2\vartheta\right)^2 + \frac{\Phi^2}{N^2}\left(\frac{dS(\vartheta)}{d\vartheta}\right)\sin\vartheta\cos\vartheta - S(\vartheta)\right]^{1/2}, \tag{1}$$

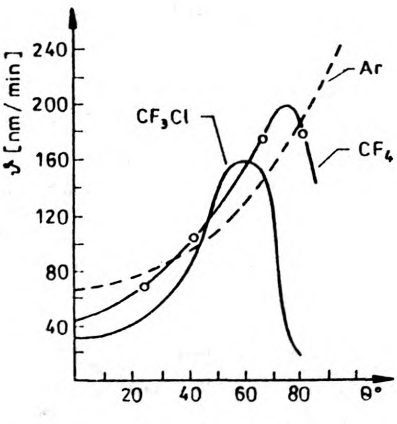
and the direction of the transfer is given by

$$(\tan\alpha)_\vartheta = \frac{v_x}{v_y}\left[\frac{dS(\vartheta)}{d\vartheta}\right]\sin\vartheta\cos\vartheta - S(\vartheta)\Big/\left[\frac{dS(\vartheta)}{d\vartheta}\right]\cos^2(\vartheta) \tag{2}$$

where: Φ – ion flow, N – number of atoms in 1 volume, S – sputtering yield of the material target, ϑ – local angle of incidence ion upon a point of the surface [7].

In the development of Carter's theory, Barber [8] shows that if the dependence $S(\vartheta)$ is known, it is possible to produce a non-normalized erosion curve, that is a polar diagram of $[S(\vartheta)\cos\vartheta]^{-1}$ dependence on the angle of ion incidence and to use it for successive analyses of either the surface morphology evolution or amorphous structures under physical sputtering in the inert gas atmosphere.

The results of etching the glass with the ions incident normally to the surface through a holographic grating of the sinusoidal profile are presented in this paper. The grating with a sinusoidal profile of spatial frequency of 1200 lines/mm was recorded on the layer of photosensitive composition by holographic method. The depth of grating relief was 0.15 – 0.29 μm, while the total thickness of photoresist layer was 0.3 – 0.5 μm.



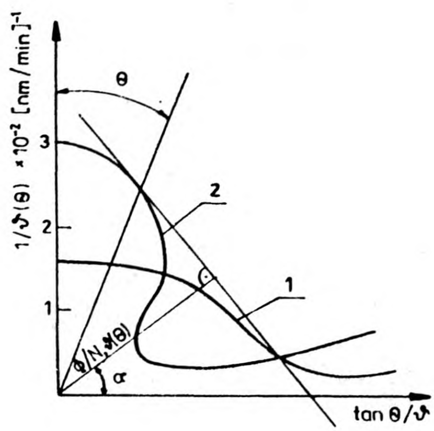


Fig. 2. Angular dependence of the etching rate of photosensitive composition on glasses Ar, CF_3Cl, CF_4

Fig. 3. Photosensitive composition of curves' erosion, treated for gases Ar (curve 1) and CF_3Cl (curve 2)

In order to determine the optimal conditions of forming the grating of symmetric and asymmetric profiles, the angular dependence of the etch rate of photosensitive composition in different gases, such as: Ar, CF_4, CF_3Cl has been investigated. The incident angle of the ions was changed within the 0 – 80° angle range (Fig. 2). As it may be seen in Fig. 2, for the inert gas Ar and active gases CF_3Cl, CF_4 and small and middle angles, the dependence $S(\vartheta) = S_0 \sec\vartheta$ can be assumed while maximal sputtering is obtained for angles between 60 – 70°, for all gases.

Following the experimentally obtained dependence of the etch rate on the angle,

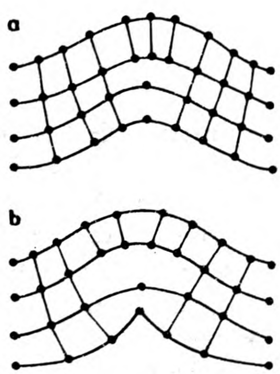


Fig. 4. Erosion of the mask profile while etched in gaseous Ar (a) and gaseous CF_3Cl (b)

the erosion curves were obtained for photoresistive composition in Ar and CF_3Cl (see Fig. 3).

With the help of the erosion curve a successful development of the surface of sputtering could be reached.

In Figure 4, the results of geometrical shape of the mask profile for equal time of etching in the Ar (curve a) and CF_3Cl (curve b) are presented.

The minimum erosion of the sinusoidal profile occurred in Ar, in chemically active gas erosion has been more serious. It can be explained if the investigated grating has $h/d = 0.24$. The angle of ion incidence measured to the initial profile is not greater than 35° (for generally normal incidence). In this range of the angles, the sputtering rate of the photosensitive composition is constant in Ar and it is changed from 35 to 55 nm/min in CF_3Cl. Under these circumstances the profile obtained on the glass in CF_3Cl has the sharppulled shape.

For the transformation of the profile of the grating patterns in the layer of the

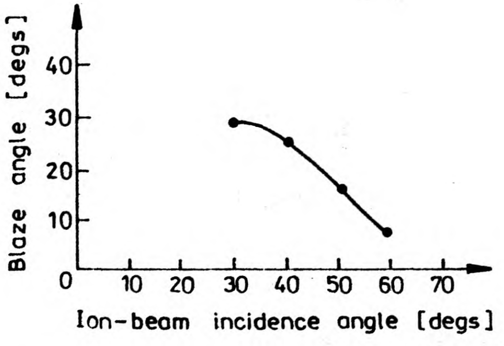


Fig. 5. Blaze angle of diffraction grating as function of angle of ion incidence

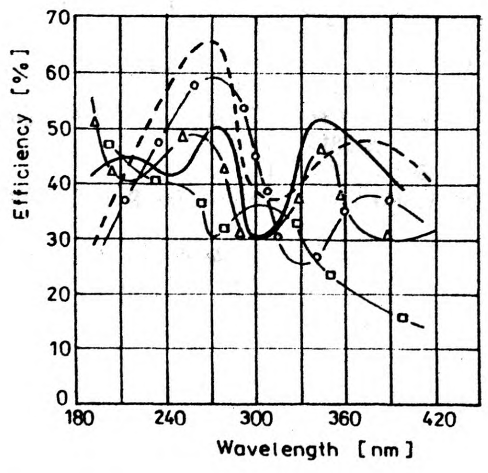


Fig. 6. Diffraction efficiency of grating produced for different angle of ion incidence (—— initial, o – 30°, – – – 40°, Δ – 50°, □ – 60°)

photoresistive composition with the help of the ion-beam, the variation of the sputtering rate versus the angle of incidence provides an important possibility.

As shown for the grating, the sinusoidal profile on the glass, the angular dependence of the sputtering rate leads to a strong degradation of the grating structure.

As may be seen in Figure 2, the sputtering rate of the photosensitive composition is constant in Ar for angles contained within 0–35° range. If the angle of the ion-beam incidence could not exceed 35°, than the appropriate gratings constant ratio d/h should be determined.

For the grating with pattern density 2400 lines/mm for $h = 0.15$ μm and $d = 0.48$ μm the angle of the ion incidence on the surface is as high as 60°.

Figure 5 shows relation between the blaze angle of etched grating and angle of ion incidence for the grating with pattern density 2400 lines/mm. Thus, a blaze angle of the grating between 9–30° can be formed at various angles of ion incidence between 30–60°. Diffraction efficiencies of etched grating in the visible and ultraviolet wavelength region were 55–65% (Fig. 6).

4. Conclusions

It has been demonstrated that the ion-beam and reactive ion-beam etching and holographic exposure constitute a powerful method for transformation of the gratings pattern profile and for holographic diffraction grating manufactured on the surface of optical glass.

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