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On the fabrication of diffraction gratings employed in integrated optics*

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The details of the experimental technique of photoresist and GaAs gratings fabrication have been presented. Different dilutions of photoresist, exposure and etching times have been examined in order to obtain the optimum conditions for experimental procedure, resulting in uniform, efficient gratings. Structures on silicon and GaAs substrate have been investigated in the work. The optimum exposure and etching times, in the case of GaAs grating fabrication, have been obtained.

1. Introduction

Integrated optics requires the production of grating structures having periods ranging from 0.1 to 5 μm. These are employed as a beam-to-wave couplers, filters, polarizers and distributed elements (see e.g. [1–3]). The wide range of periods, aperture, modulation depth, together with the requirement of high degree of uniformity creates specialized problems during photolitographic processing.

Depending on the application, the final grating may be fabricated in the resist itself, the resist may serve as a mask for chemical etching, sputter etching or ion milling, or it can act as a mask for further deposition or ion implementation [4]. Regardless of its use, a good controll over the grating pattern is necessary to produce device with the desired characteristics.

In the present paper, two types of the gratings have been investigated. One, formed in the photoresist on Si and GaAs substrates and the second formed be means of chemical etching in GaAs substrate. The results presented show the dependence of the diffraction efficiency of the grating and its uniformity on the dilution of the photoresist, exposure and etching time. Reported information should be useful in the fabrication of efficient gratings, both formed in photoresist and etched in the substrate material.

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2. Fabrication of the gratings

The general experimental procedures in fabrication of the resist grating by holographical means, employing Ar laser have been described in several papers (see e.g. [4, 5, 6]).

For the investigation, Shipley AZ 1350 was used as a photoresist. It has peak sensitivity in ultraviolet and the use of argon laser results in log exposure times. It has been shown that 457.9 nm output, though of reduced power compared with that obtained at 488.0 nm, is more effective. Therefore, throughout this work, the 457.9 nm output from argon laser has been used. Its output power was about 100 mW, what resulted in power density of about 2 mW/cm^2 in the image plane. A beam expander telescope employed in the interferometer system extended the laser beam to approximately 1 cm in diamteter in the image plane. Extreme care in the preparation of the substrates was necessary, particularly of those made of silica. Crazing of the resist occurs during its development if precautions are not taken to derive all the traces of water from the substrate surface [5]. Therefore, after cleaning and rinsing in deionized water the substrates were given a bath in ethyl alcohol followed by a bake at 373 K for 2 hours. In the case of Si two dilutions: 1 : 1 and 2 : 1 of Shipley AZ 1350 have been used. The photoresist layer was produced by spin coating at 6000 rpm for 30 sec. Different exposures ranging from 70 mJ/cm^2 to 140 mJ/cm^2 were examined. After the exposure for the laser beam forming the grating in the layer the resist was developed in 1 : 1 diluted AZ Developer at R. T. for 1 minute. In the case of fabrication of the grating in GaAs substrate, before the photoresist deposition, the crystal walls of GaAs were obtained according to the method presented in [7, 8]. When the ⟨100⟩ plane is etched, well defined V-shaped-grooves can be obtained. Such a shape yields well defined, efficient structures. After spin coating, followed by the exposure and development (in the way described above) the obtained photoresist grating was used as a mask. The $H_2O_2 : NH_4OH$ (1 : 3) etching system was applied at 293 K so that Shipley AZ 1350 resist could be directly used as a protective mask during chemical etching. Diffraction efficiency during investigations was obtained by means of Helium-Neon laser beam incident normally on the grating. The intensity of the first order beam from the grating relative to the incident beam was the measure of diffraction efficiency.

3. Characteristics of photoresist grating

In fabrication of gratings, the control over the initial resist thickness and residual resists (obtained after exposure and development) is important. Scattering and other losses in grating couplers can be minimized by avoiding a residual layer. When employing the grating as a mask for sputter or chemical etching or ion milling, the photoresist grating pattern should extend completely down to substrate surfaces. By differentiating thickness of the initial resist layer, exposure and developing time one can obtain layers of different thickness, depth and shape of modulation.

In the experiment with the photoresist grating on Si substrate, 1 : 1 and 1 : 2 diluted Shipley AZ 1350 have been employed. The initial layers thickness were about 0.10–0.2 μm, respectively. As a second variable different exposures ranging from 70 to 140 mJ/cm² were studied. They yielded underexposed, properly exposed and overexposed gratings. As a measure of the grating uniformity, the diffraction efficiency of each structure has been measured for each exposure. The properly exposed structures show high efficiency in the range of exposure of 90–125 mJ/cm², being in good agreement with the results of [9] (fig. 1). Both overexposed and underexposed curves result in lower diffrac-

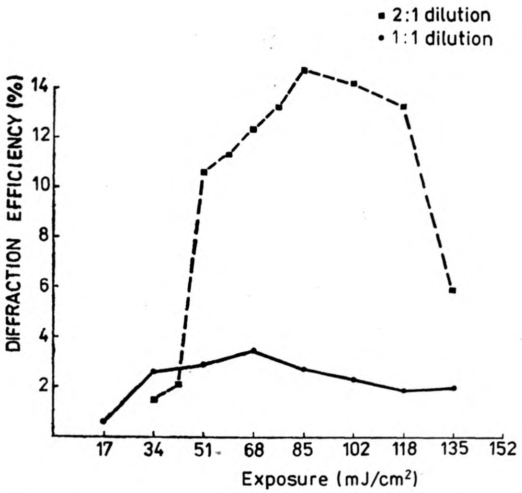


Fig. 1. Diffraction efficiency vs. exposure for 1 : 1 and 2 : 1 (ratio of AZ 1350 to thinner) layers on Si substrate

tion and uniformity. An analogical curve for 1 : 1 diluted Shipley AZ 1350 (fig. 1) shows a lower diffraction efficiency for almost all exposures. More regular, deep grooves obtained for the thicker layer yield higher diffraction efficiency. Figure 2 shows the diffraction efficiency vs. exposure characteristics for gratings fabricated on Si and GaAs substrates.

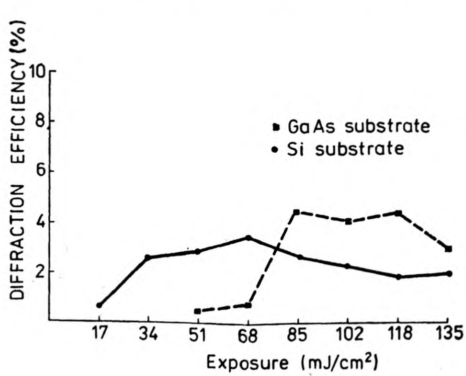


Fig. 2. Diffraction efficiency vs. exposure for photoresist layer (dilution 1 : 1) on Si and GaAs substrates

4. Optimization of the photoresist exposure and etching time in GaAs substrate grating fabrication

For the maximum diffraction efficiency and high quality of GaAs grating an accurate exposure of photoresist during the interferometric delineation of diffraction grating pattern is essential. However, the optimum exposure is a strong function of absorption and reflection properties of the wavelength employed [4]. The surface damage and possible oxide layers have to be also considered, since they modify the substrate absorption and reflection. The optimal exposures are, therefore, material-dependent and exact numerical prescriptions are difficult to quote. Only empirical approach with some general guidelines can supply means for obtaining right exposure for various substrates [4]. Figure 3 shows experimental curves relating the diffraction efficiency of

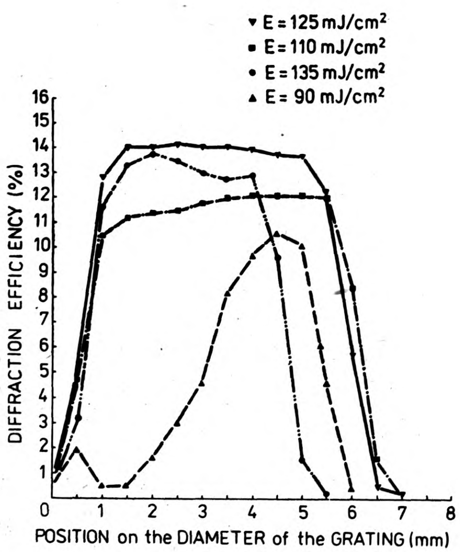


Fig. 3. Diffraction efficiency vs. position on the diameter of the grating at four selected exposures

the GaAs grating to the position on the diameter of the grating pattern at different exposures. The exposure of about 125 mJ/cm² results in efficient gratings. The best uniformity of the structure has been obtained also for 125 mJ /cm² exposure. For the maximum diffraction efficiency and quality of GaAs grating the accurate etching time is also essential. Figure 4 shows three experimental curves relating the diffraction efficiency of the GaAs grating to the position on the diameter of the grating pattern, at three etching times. According to the presented above results 125 mJ/cm² exposure has been applied. The highest (more than 14%) diffraction efficiency and uniformity have been obtained for the case of one minute etching time. The photograph of the photoresist grating mask and SEM photograph of the adequate grating in the substrate, obtained for the optimum exposure and etching time, are shown in figs. 5a and 5b, respectively.

In conclusion, the properties of the photoresist and adequate GaAs grating have been compared. Experimental curves relating the diffraction efficiency of the photoresist grating and GaAs grating to the position on the diameter

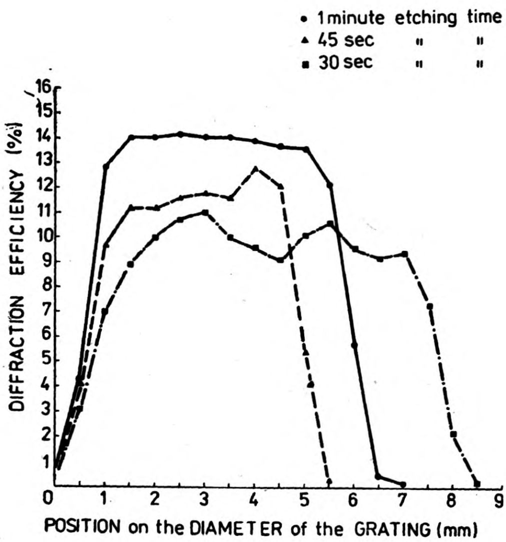
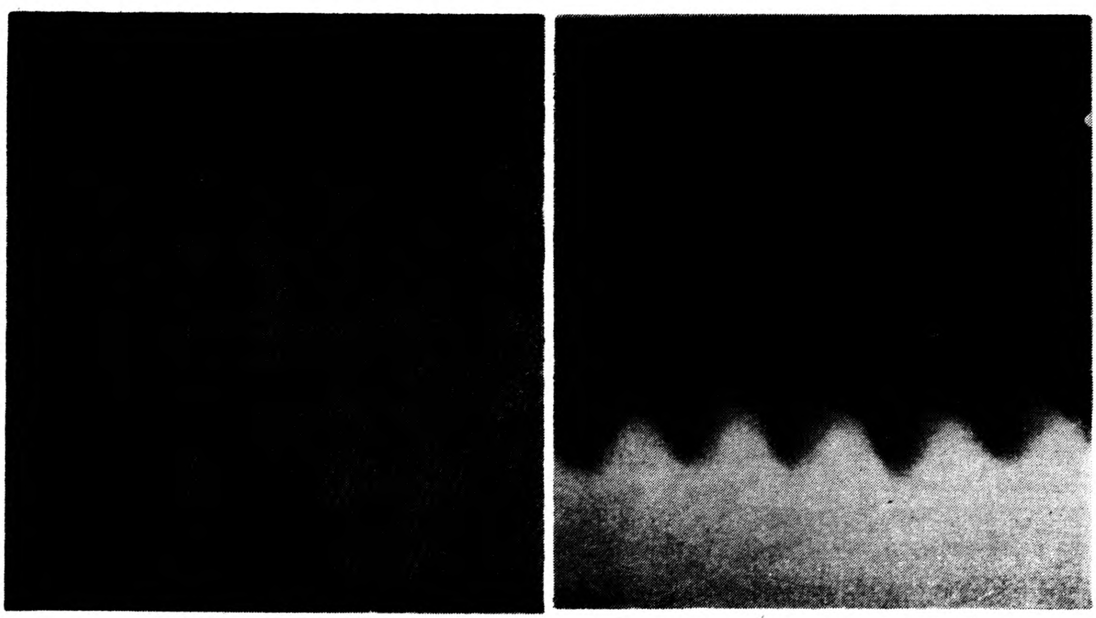


Fig. 4. Diffraction efficiency vs. position on the diameter of the grating for three etching times



a b

Fig. 5. a. Optical photograph of the part of grating made in photoresist, b. scanning electron photograph of the GaAs grating

of grating pattern obtained for exposures of about 100, 120, 135 Jm/cm² are presented in figs. 6a, 6b, and 6c, respectively. Note, that the exposure which corresponds to the efficient photoresist grating of about 120 mJ cm² (being in

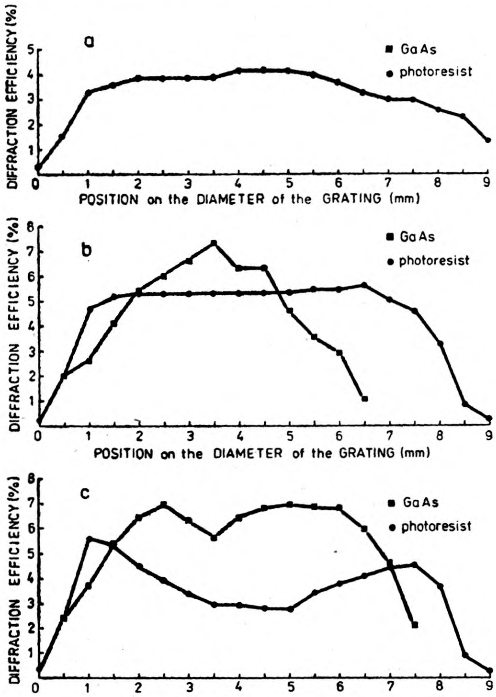


Fig. 6. Diffraction efficiency of photoresist and GaAs gratings vs. positions on the diameter of the grating at 100 mJ/cm² (a), 120 mJ/cm² (b), and 135 mJ/cm² exposures (c)

good agreement with the results of [9]), does not correspond to the optimum exposure for GaAs grating. The photoresist mask should be slightly overexposed, hence it would uncover the vider area of the surface, necessary for a suitable depth and profile of etched gratings.

5. Conclusions

Two types of gratings, formed in photoresist and etched in substrate material have been studied. Some characteristics of photoresist grating, obtained for different dilutions of Shipley AZ 1350 and placed on Si and GaAs substrates, are reported. By employing different exposures and etching times the optimum conditions for photoresist garting mask and GaAs etched grating fabrication have been obtained. For the case of photoresist grating on the GaAs substrate, investigated in this work, the exposure of about 110–125 mJ/cm² assures the efficient uniform structures. However, despite the overexposed photoresist, the 135 mJ/cm² exposure yields the best (of the highest diffraction efficiency) final substrate grating. Such an exposure and preferential one minute etching results in fine, uniform GaAs grating. In that case diffration efficiency was better than 14%.

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Изготовление дифракционных решёток, применяемых в интегрированной оптике

Дано подробное описание экспериментальной техники изготовления решёток GaAs при использовании фоторезистов. Были исследованы различные растворы фоторезистов, различные количества освещения, а также продолжительности травления для получения оптимальных экспериментальных условий, в которых были бы получены однородные и производительные решётки. Исследовались структуры на силиконовых подложках и из GaAs, а также решётки, образуемые внутри подложки из GaAs. Были получены оптимальные количества освещения, а также продолжительности травления для случая решёток из GaAs.