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PHYSICAL AND ENGINEERING FUNDAMENTALS
OF MICROELECTRONICS AND OPTOELECTRONICS

Silicon Diffractive Optical Elements for High-Power Monochromatic Terahertz Radiation

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Abstract—This paper presents a fabrication technique and results of studies of silicon binary diffractive optical elements (DOEs): a diffractive lens and a 1 : 2 diffractive beam splitter with an aperture diameter of 30 mm for the terahertz spectral range. The elements were fabricated in two versions: with and without an antireflection coating of parylene *C*. The DOE characteristics were investigated in the beam of the Novosibirsk free electron laser at a wavelength of 141 μm. The results are given of a study of the radiation resistance of the coating, which remained intact upon exposure to an average radiation power density of 4 kW/cm^2; the peak power in a 100 ps pulse was almost 8 MW/cm^2. Experimental estimates of the diffraction efficiency of the elements coated with the antireflection coating are in good agreement with theoretical estimates.

Keywords: DOE, terahertz radiation.

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INTRODUCTION

Diffractive optical elements (DOEs) have found wide application in laser processing and optical devices of the ultraviolet, visible, and infrared ranges [1]. DOEs can be used to design optical devices with reduced weight and size and wide functionality. Due to the higher (compared to the visible range) absorption of materials transparent in the terahertz range, DOEs are preferred over refractive elements (lenses, prisms). Diffractive optical elements have little alternative in the case of controlling the monochromatic beam of a high-power free electron laser (FEL) [2]. Applications such as terahertz imaging, soft ablation, optical discharge, and many others call for focusing of terahertz radiation. Other applications, such terahertz holography [3], interferometry and polarimetry, require the solution of dividing the original beam into several spaced beams with a given distribution of energy between them.

A technology of fabricating diffractive lenses from solid polypropylene by hot pressing is described in [4]. It has been shown [4, 5] that they can be used in high-power terahertz beams to produce images with a resolution close to the diffraction limit. The technology is simple and allows producing diffraction lenses with different specified characteristics. However, there is a practical need to design DOEs with a specific spatial distribution of power, which requires the production of complex spatial reliefs [1]. In such cases, it is more promising to use high-resistivity silicon, on the surface of which fine structures of various shapes can be produced using modern technology.

The objectives of this work were to improve the technology of producing microrelief on the surface of high-resistivity silicon wafers, and to design and fabricate two types of DOEs, with subsequent application of an antireflection coating on them, and to test the diffractive elements using a powerful terahertz radiation source. The obtained experimental results are in good agreement with theoretical calculations. Thus, it has been demonstrated that DOEs with a parylene coated in the terahertz range have high radiation resistance and can therefore be used to control a high-power free electron laser.

TECHNOLOGY OF PRODUCING A BINARY DIFFRACTIVE SILICON RELIEF OF THE TERAHERTZ RANGE

If undoped high-resistivity silicon is used as the substrate material for terahertz DOEs designed to control high-energy beams (for example, radiation FEL), impurities do not reduce the transmittance of the wafer. In the present study, we used substrates of HRFZ-Si silicon [6], with two-sided polishing of optical quality 100 mm in diameter and 1 mm thick. Silicon DOEs were fabricated jointly with the Samara State Aerospace University, Image Processing Systems Institute of RAS, and Tydex company. The fabrication route of the DOEs is shown in Fig. 1.

The height of the diffractive microrelief of the binary DOE is determined from the formula [1]

$$h = \lambda/2(n-1), \tag{1}$$

where n is the refractive index of the substrate material and λ is the wavelength of the illuminating light. Microrelief of great height (about 27 μm, $\lambda = 130$ μm and $n = 3.41$ was produced by reactive ion etching (RIE) [7]. Due to the low plasma resistance of the photoresist mask, it cannot be used without an additional masking layer. Therefore, in this paper, we use plasma-resistant metal masks of copper and aluminum. The masks were produced by forming windows in the thin metal film deposited on the silicon surface by photolithography (using a FP-4-04mA positive photoresist, providing resolution not worse than 0.5 μm) followed by chemical etching and RIE of the silicon substrate through these windows. The metal film was applied using an ETNA-100-PT device (NT-MDT company, Russia).

In the development of the technology, it was found that the copper mask has an order of magnitude higher selectivity in the RIE process compared to an aluminum mask (>1 : 300). In addition, the copper foil has poor adhesion to silicon, so that before applying a copper coating, it is necessary to apply an adhesive sublayer. We used a chromium sublayer 30 nm thick. A topological pattern in the copper layer was produced by liquid etching through the photoresist mask in a 5% solution of ferric chloride ($FeCl_3$). Thereafter, the

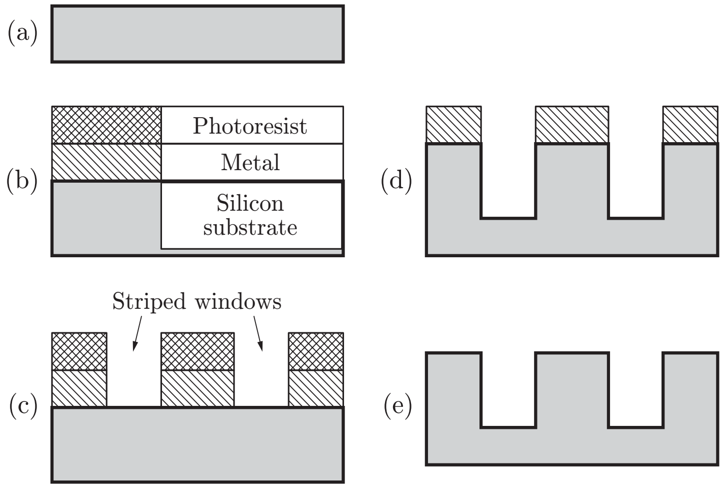

Fig. 1. Fabrication steps of DOEs: (a) silicon wafer preparation (washing and control of parameters), (b) application of metal and photoresist layers, (c) exposure of the photoresist and wet etching of the underlying metal layer, (d) reactive ion etching of silicon, (e) removal of the metal mask.

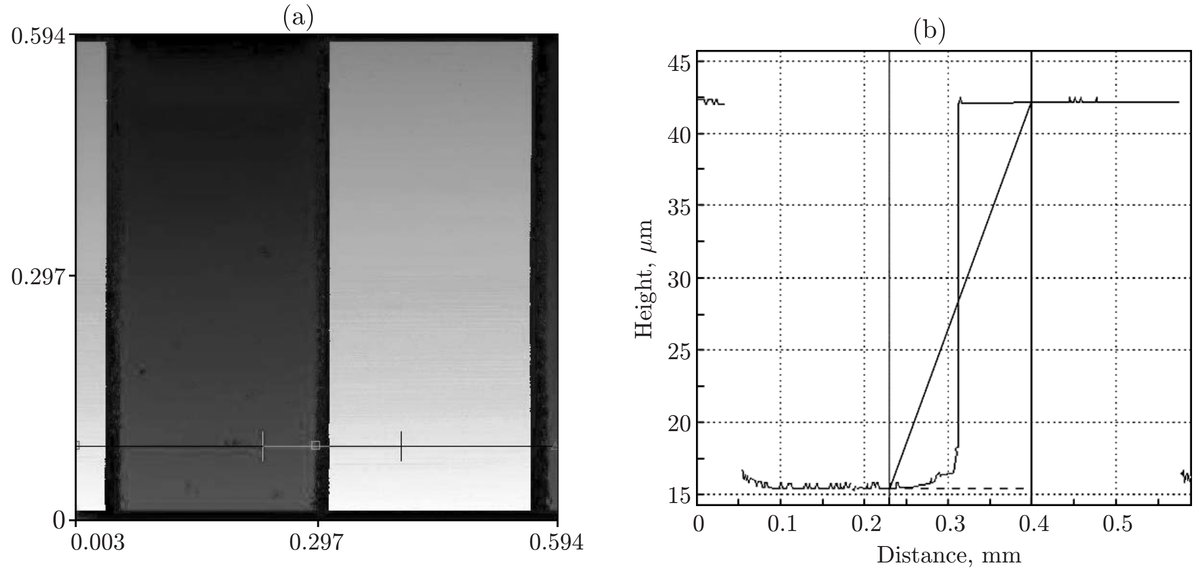

Fig. 2. Results of study of the DOE microrelief using white light interferometry: (a) photograph of a microrelief region, (b) etch depth profile along the line shown in Fig. (a).

mask was removed with concentrated alkali. The chromium underlayer was etched through the copper mask using a solution of $K_3[Fe(CN)_6] : NaOH : H_2O$ (1 : 3 : 16).

The aluminum film has a high adhesion to silicon, eliminating the need to use an adhesive underlayer. Etching of the aluminum film was made immediately after the development of the photoresist mask, in the same developer, without removing the sample. High etch rate provides preservation of the photoresist mask and full and high-quality etching of aluminum. Thus, an advantage of this material is a much simpler and faster production of a mask.

Production of microrelief in silicon by RIE for fabrication of terahertz DOEs is considered in [8]. In this work, etching of silicon performed on an ETNA-100-PT device (NT-MDT, Russia). To achieve the specified performance of the DOEs, it was necessary that the angle between the walls and the vertical be not more than 10°. Therefore, we used the Bosch process in an inductively coupled plasma source configuration (ICP–RIE) [9] in an atmosphere of C_4F_8/Ar (passivation) and SF_6/Ar (etching). The parameters were chosen to provide optimum etch rate (at a level of 1 μm/min), a small angle of deviation from the vertical (less than 5°), slight etching under the mask (variation in the width of the elements not more than 5 μm), and acceptable quality of the surface of the walls. The reactor pressure was about 0.1 mm Hg and depended on the stage of the etching cycle. The reactor was evacuated by a turbomolecular pump, which provided fast renewal of the reaction atmosphere. In all cycles of etching, an ion-forming additive of inert argon gas (20 l/h) was used. The consumption was C_4F_8 was about 60 l/h, and that of SF_6 about 30 l/h The ICP source of the inductor had a power of 300 W and a frequency of 13.56 MHz. The accelerating source only operated in the etching stage and only at a constant DC-bias of 180 V; its power did not exceed 25 W, and the source frequency was 13.56 MHz. The duration of the cycles was chosen so as to provide the verticality of the walls and was 11 s for the passivation phase and 9 s for the etching phase. The etch depth for one cycle was 350 nm. The total etch depth was specified by the number of cycles. After the cyclic etching, the surface was subjected to short-term cleaning in an SF_6 atmosphere in the isotropic etching regime.

The geometric parameters of the microrelief were controlled with a WLI-DMR white light interferometer produced at the Fraunhofer Institute for Production (Jena, Germany) (Fig. 2) and a Quanta-200 scanning electron microscope (FEI corporation).

The elements were fabricated in two versions: with and without an antireflection coating of parylene *C* (polyparaxylene). Parylene *C* as an antireflection coating was used in [10, 11].

BINARY DIFFRACTIVE LENSES

In this work, we fabricated binary diffractive lens with a focal length of 120 mm and an aperture diameter of 30 mm for a wavelength of 130 μm (Fig. 3).

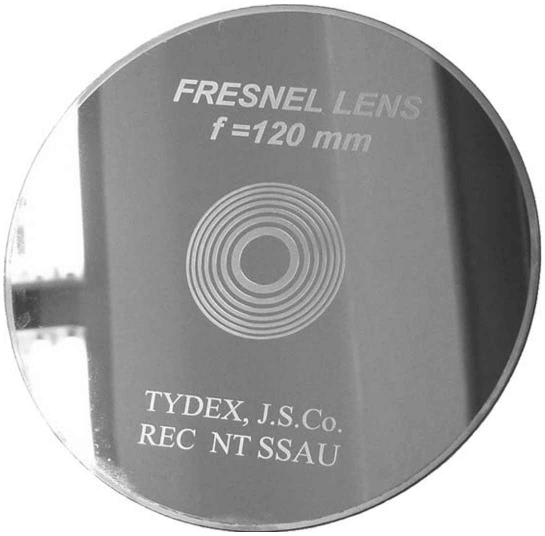

Fig. 3. Appearance of the binary diffractive lens.

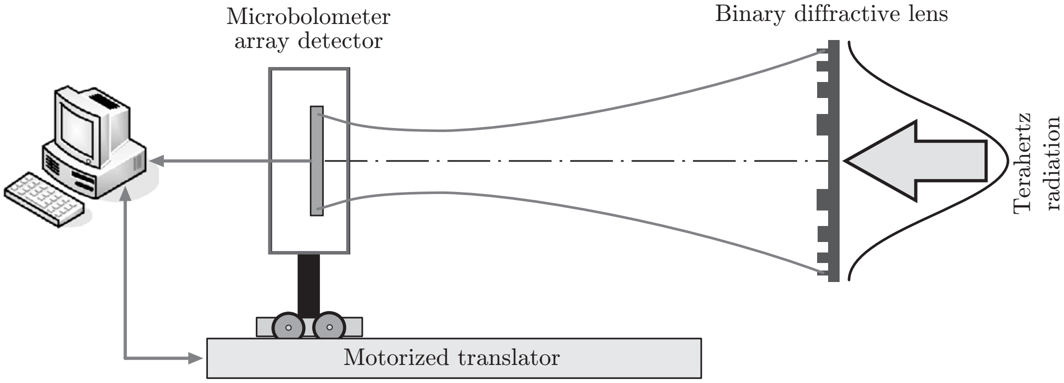

Fig. 4. Optical scheme of the study of the binary diffractive lens on the FEL.

The optical characteristics of the lens were examined on one of the FEL workstations (Fig. 4). The laser generated monochromatic radiation with a pulse duration of 100 ps at a repetition rate of 5.6 MHz. The laser beam had a Gaussian distribution $I = I_0 \exp(-2r^2/w^2)$, where $w = 9$ mm, i. e. almost 100% of the energy of the beam passed through the element aperture with a diameter of 30 mm. The average radiation power in the experiments was a few watts. A binary diffractive lens was designed and constructed to focus the radiation at a distance $f = 120$ mm at a wavelength of 130 μm. In the experiments, the minimum laser wavelength was bounded by $\lambda = 141$ μm, at which all experiments were performed. For this wavelength, the focal length, according to theory, should be 110 mm instead of 120 mm. The radiation transmitted through the element was detected by a a microbolometric array detector of 320×240 elements (physical size of 12.24×16.36 mm) [12] which was moved with a motorized translational table along the optical axis.

Two focal distances at distances of 110 and 42 mm from the lens (Fig. 5) were observed, which, in view of the measurement accuracy of about ±3 mm, is in good agreement with the simulation results. The diffraction efficiency of the lens without the anti-reflection coating was $(21 \pm 3)\%$ for the main focus, and 3% for the secondary focus, and for the lens with the antireflection coating, $(36 \pm 5)\%$ and 3.6%. The estimate of the diffraction efficiency is in good agreement with the theoretical diffraction efficiency of the binary diffractive lens — 41% [1].

Figure 6 shows the intensity distribution in the focal spots of the diffractive lenses in comparison with the focus of the TPX lens (polymethyl pentene) [5].

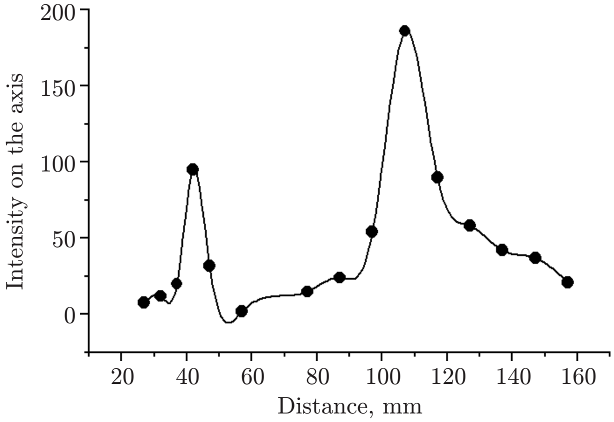

Fig. 5. Axial intensity distribution formed by the binary diffractive tional lens.

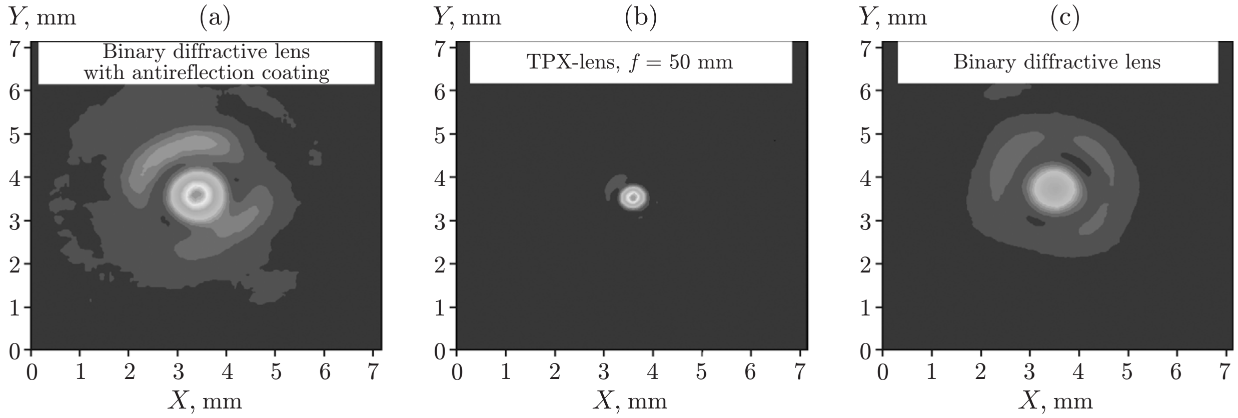

Fig. 6. Intensity distribution in the focus.

Tests were performed of the radiation resistance of the diffractive lens with the antireflection coating. For this, the peripheral part of the silicon wafer with the antireflection coating (outside the diffractive structure) was irradiated by the terahertz radiation of the FEL focused by a TPX lens [5]. The absolute values of the power density distribution were measured with a thermosensitive interferometer [13]. The lenses were not damaged up to a power density of 4 kW/cm^2 at the maximum of the Gaussian distribution, which corresponds to a peak power of about 8 MW/cm^2 for a 100 ps pulse.

1 : 2 BINARY DIFFRACTIVE BEAM SPLITTERS

Binary diffractive beam splitters with an aperture diameter of 30 mm were designed, fabricated, and studied. The diffraction microrelief of the beam splitter was a grating with a rectangular profile and a period of 500 μm. The optical characteristics of the beam splitters were also analyzed on one of the FEL workstations (Fig. 7). The terahertz radiation from the transport channel of the FEL filled with nitrogen was directed to the workstation. The radiation power was controlled by two polarizers (since the FEL radiation is linearly polarized). The radiation wavelength in the experiments was also 141 μm. For continuous control of the FEL radiation power, the reference beam extracted with a polypropylene film beam splitter was sent to a GC-1T Golay cell (Tydex [6]) with a SR830 synchronous amplifier, because in these experiments, the FEL radiation intensity fluctuated within ±5% during measurements. The same microbolometer array detector was used as the detector. Since the beam diameter was 30 mm, it was possible to completely intercept it on the array only with a lens (TPX lens with a focal distance of 200 mm). The beam splitters were placed at a distance of about 3 mm from the lens. For correct measurements of all diffraction orders (the ±1st orders are incident at an angle of 15°), the microbolometer array was moved radially, so that the rays were normally incident on the array.

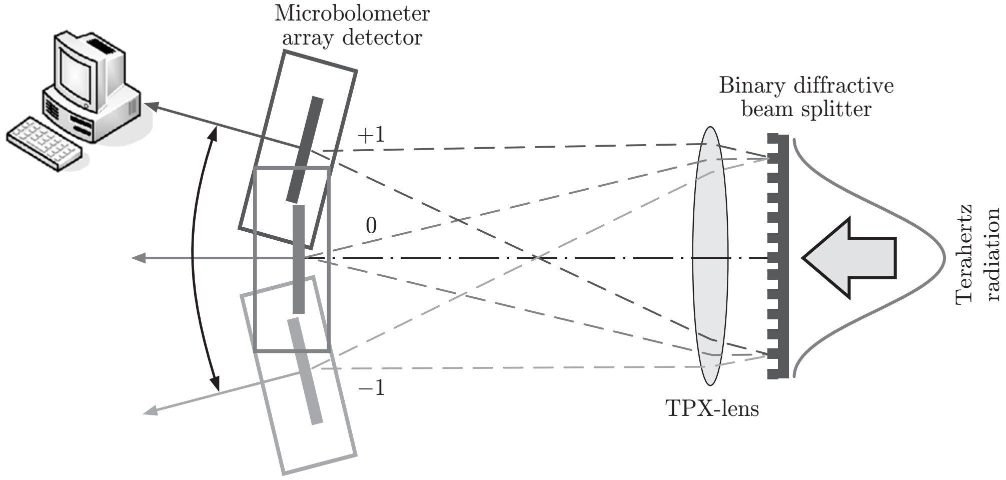

Fig. 7. Optical scheme of the study of the binary diffractive beam splitter on the FEL.

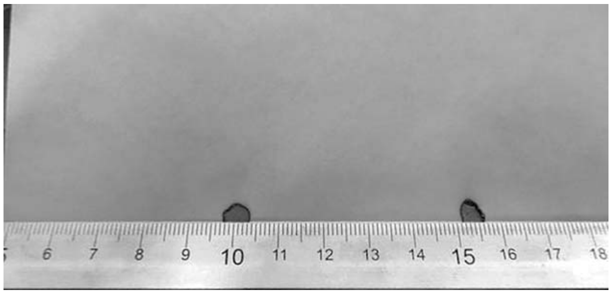

Fig. 8. Result of interaction of terahertz radiation with paper in the ±1-st diffraction orders of the beam splitter.

The experimental estimate of the total fraction of the energy of the incident beam diffracted to the ±1-st orders was (50 ± 5) % for the beam splitter without the anti-reflection coating and (79 ± 8) % for the beam splitter with the antireflection coating. The obtained experimental estimate of the diffraction efficiency of the beam splitter is in good agreement with the theoretical estimate of the diffraction efficiency of the 1 : 2 binary diffractive beam splitter — 81% [1]. At the same time, there was a slight difference between the fractions of the energy per the ±1-st diffraction orders (the relative difference was about 25% for the element with the antireflection coating) which may be explained by inaccuracy in the production of the microrelief. Figure 8 shows the holes burned through by the FEL beam in writing paper placed in the focal plane of the TPX lens for one second. It is seen that almost the whole energy goes to the ±1-st diffraction orders.

CONCLUSIONS

The experiments described in this paper show that the technology of reactive ion etching of high-resistivity silicon with subsequent application of the antireflection coating of Parylene *C* is suitable for fabricating diffractive optical elements for controlling powerful terahertz radiation. The obtained experimental results are in good agreement with available theoretical results and offer hope for designing terahertz DOEs generating a specified two-dimensional intensity distribution — focusers of the terahertz range. The DOEs with a parylene coating have high radiation resistance in the terahertz range and can be used to control high-power free-electron laser radiation. The diffraction efficiency of the elements can be enhanced by improving the fabrication technology, in particular, by increasing the number of quantization levels of the microrelief [1],

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