

Features of the mesa-structure formation via electron-beam lithography in semiconductor GaAs-based compounds

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We study wet chemical etching of the low-temperature grown GaAs (LT-GaAs) using the resistive mask produced with electron-beam lithography. Efficient etching of the mesa-structure (i.e. mesa) is developed allowing to control exact parameters of the mesa in the given areas of LT-GaAs. The technology includes an essential retreatment of the LT-GaAs surface before applying the resist to improve adhesion, as well as treatment in oxygen plasma after its development. Applying the proposed technology, we fabricated large-area photoconductive THz emitter with sophisticated topology of the mesa-structure.

Keywords: optoelectronic source, terahertz radiation, wet chemical etching, mesa structure, semiconductors, electron-beam lithography.

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The onset of active development of the terahertz (THz) range of the electromagnetic spectrum is associated with the advent of pulsed THz spectroscopy (spectroscopy with recording of the shape of THz pulses) [1]. Various physical principles (e.g., nonlinear phenomena) may be utilized in this method to generate and detect THz pulses; however, owing to their simplicity and reliability, photoconductive (optoelectronic) sources and detectors excited by femtosecond laser pulses are the ones used most often [2,3]. However, since the efficiency of conversion of the laser excitation pulse energy into THz radiation is rather low, individual optoelectronic devices require specific (and often quite expensive) design solutions, including plasmonic or interdigital electrodes, dielectric metasurfaces, etc. [3]. At the same time, the use of multi-element optoelectronic sources consisting of a one-dimensional or two-dimensional matrix of unit emitters is an efficient way to increase the THz generation power [4,5]. The key problem in shaping the topology of a multi-element source is the need for electrical insulation of adjacent elements [6], which helps reduce the parasitic (dark) current significantly and prevent the formation of a pattern of destructive interference of THz radiation from in-phase unit sources in the far field. To form such insulation, one needs to etch elements of a mesa-structure in the photoconductive layer regions between individual topology elements.

In the present study, we propose an original method for creating a mesa-structure with precisely reproducible dimensions based on a mask formed by electron-beam lithography (EBL) that is used for wet chemical etching (WCE) of a GaAs layer grown by molecular-beam epitaxy at a low temperature (low-temperature-grown GaAs, LT-GaAs). Note that EBL has virtually no alternatives when one needs to work with small-area samples or when high

accuracy in the sizes and positioning of mask elements is required.

The method of plasma-enhanced chemical etching with chlorine-containing gases is commonly used to etch mesa-structures [7]. In this case, the pattern for etching is formed by photolithography using a resist that is resistant to plasma exposure. If a complex multi-element topology is to be formed, the positioning and size of the mesa-structure etching regions must be specified with high accuracy; therefore, EBL was used to form the pattern. Compared to photoresists, the electron resist used in this case has lower a plasma resistance. This necessitates the use of WCE, since a considerable mesa-structure etching depth within the range of $1-2\mu\text{m}$ is needed. Wet etching with a mask formed by EBL has its own specifics that must be taken into account to achieve a high-quality result. Specifically, the profile of etching regions in WCE may have characteristic undercut (caused by selectivity with respect to crystallographic planes; see Fig. 1, a) and under-layer etching (etching under the resist due to its delamination as a result of loss of adhesion; Fig. 1, b) features. Since under-layer etching is a negative effect that changes the size and quality of the element boundaries, it needs to be minimized

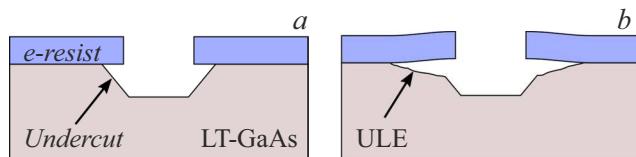


Figure 1. Schematic diagram of the mesa-structure profile features formed during WCE in an LT-GaAs layer with a mask formed by EBL. a — Etching in both directions (undercut); b — etching under the resist due to its delamination as a result of poor adhesion (under-layer etching, ULE).

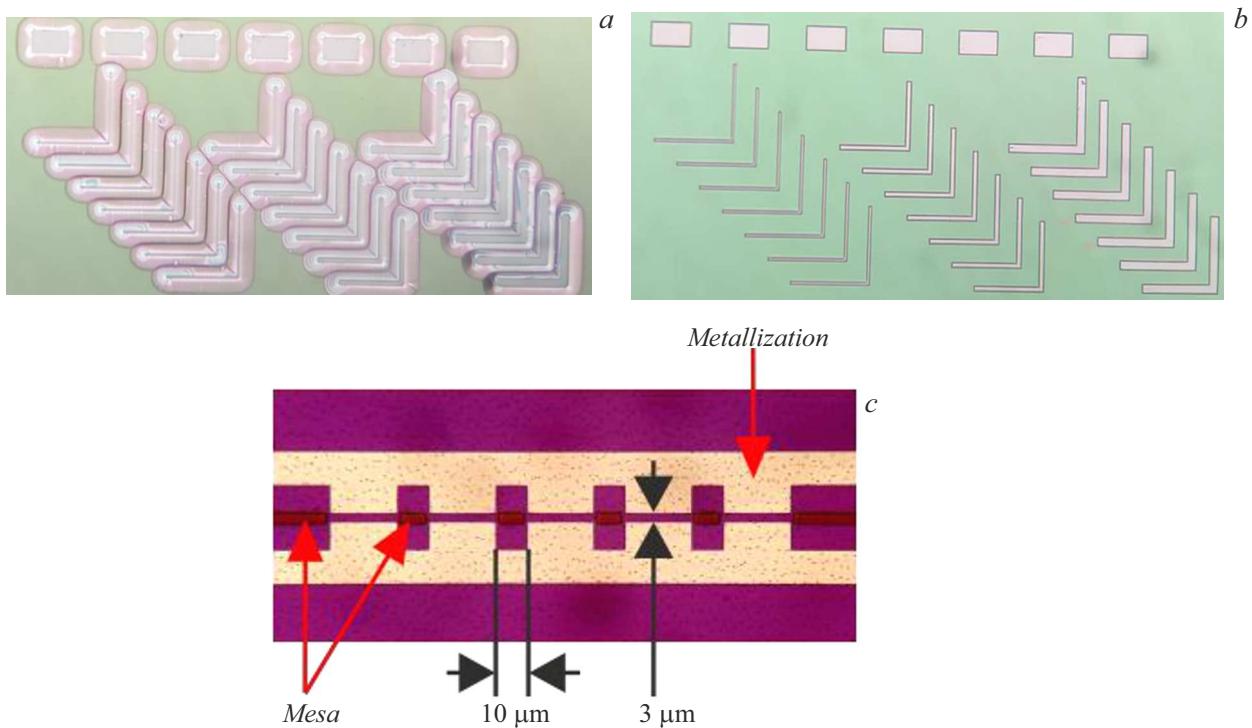


Figure 2. Optical microscope images illustrating the results of etching of elements with a PMMA 950K A4 resist mask. *a* — Test elements without pretreatment; *b* — test elements with pre- and posttreatment; and *c* — enlarged image of a fragment of topology of a five-element optoelectronic THz source having a complex mesa-structure (mesa) with its element sizes indicated.

or eliminated completely in order to ensure that the etched pattern matches the original.

LT-GaAs with a thickness of $1.5\text{ }\mu\text{m}$ formed on a GaAs substrate with a crystallographic orientation in the (100) plane was used for the studies. Prior to exposure, a uniform layer of PMMA 950K A4 resist with a thickness of $\sim 0.5\text{ }\mu\text{m}$ was deposited using an ELL Raith 150 TWO setup with an accelerating voltage of 30 kV. Exposed topologies were developed in a MIBK:isopropanol (1:3) solution, and a solution based on $\text{H}_3\text{PO}_4:\text{H}_2\text{O}_2:\text{H}_2\text{O}$ in a ratio of 1:1:8, which is characterized by the variation of etching rates with crystallographic direction [8], was used for subsequent WCE. The test topology was a set of narrow bands ($100\text{ }\mu\text{m}$ in length and 2, 5, and $10\text{ }\mu\text{m}$ in width) and rectangles $50 \times 30\text{ }\mu\text{m}$ in size with the exposure doses varying in steps within the $160\text{--}245\text{ }\mu\text{C}/\text{cm}^2$ range for each element.

It was established experimentally that the lack of pretreatment of the LT-GaAs surface before the resist deposition results in delamination of the resist during etching, leading to under-layer etching for all elements at all doses, which is seen clearly in Fig. 2, *a*. Pre-treatment in an $\text{HCl}:\text{H}_2\text{O}$ (1:5) solution for 1 min at a temperature of $21\text{ }^\circ\text{C}$ was introduced in order to remove surface oxides and improve adhesion. This helped reduce the ULE depth to a value smaller than 100 nm , which is less than 10% of the LT-GaAs layer thickness and is perfectly acceptable when this material is used in optoelectronic THz radiation sources. The

lateral etching magnitude was less than $0.5\text{ }\mu\text{m}$, which is also acceptable. The next detected effect is related to the fact that etching did not proceed in resist windows exposed to the maximum dose of $245\text{ }\mu\text{C}/\text{cm}^2$. This may be attributed to local negativization of the PMMA resist [9], which is commonly associated with area doses greater than $1\text{ mC}/\text{cm}^2$. However, with small electron beam sizes (1–3 nm), the dose is distributed over a very small area with high density and a part of the resist is left undeveloped. To solve this problem, the resist (after development) was subjected to 4 min of oxygen plasma processing performed using a YES-G1000 setup (in the „electron-free plasma“ electrode configuration) with a 40 kHz capacitive discharge under an oxygen pressure of 160 mTorr and a power of 100 W , which corresponds to an approximate resist etching rate of $80\text{--}90\text{ }\text{\AA}/\text{min}$ and resulted in removal of the residual negativized resist. It should be noted that the hypothesis of local negativization is verified by the results of experiments where the etching mask pattern was also exposed to a defocused electron beam: WCE proceeded smoothly without the above-mentioned posttreatment in oxygen plasma. It was determined as a result that both pretreatment of the surface (in an $\text{HCl}:\text{H}_2\text{O}$ (1:5) solution) before the deposition of the resist and posttreatment (in oxygen plasma) after its development are necessary. These procedures made it possible to obtain a pattern of test elements etched uniformly with all topologies at all exposure doses, which is seen clearly in the micrograph (Fig. 2, *b*).

Figure 2, c shows a fragment of topology of a five-element optoelectronic THz source based on LT-GaAs, where the formed mesa-structure elements are clearly visible between its adjacent electrodes and the characteristic dimensions are indicated. The etching depth of the mesa-structure and its width were ~ 1.5 and $3 \mu\text{m}$, respectively, and the size of the mesa-structure element between individual emitters was $3 \times 6 \mu\text{m}$. Note also that the discussed method of forming a mesa-structure was applied successfully to InGaAs/InAlAs superlattice heterostructures with different molar fractions of indium grown on GaAs substrates.

Thus, the specific EBL-related features of WCE of a photoconductive LT-GaAs layer with an electron resist mask were investigated. Examined were the techniques of etching with a PMMA resist mask applied without pretreatment of the substrate and with a mask that did not undergo posttreatment in oxygen plasma after development. It was demonstrated convincingly that pre- and posttreatment procedures are necessary, since the former remove surface oxides that reduce resist adhesion, while the latter remove resist residues in the developed regions, facilitating the interaction of the etching solution with the LT-GaAs surface. Effective and reproducible principles of WCE allowing for highly uniform formation of a mesa-structure of complex geometry with an electron resist mask were outlined. Such etching technique is crucial for multi-element optoelectronic THz sources with variable topology of individual emitting elements.

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Conflict of interest

The authors declare that they have no conflict of interest.

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