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## Study of optical resistance of a bulk $\beta$ -Ga<sub>2</sub>O<sub>3</sub> crystal

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### ABSTRACT

The optical resistance and geometric parameters of the damaged region under the action of laser radiation on bulk  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> crystal were studied and calculated depending on the power and frequency of laser irradiation. The sample of the crystal for the study was grown by the Czochralski method and prepared by method of cleaving along the (100) plane. The optical resistance was calculated by the Liu method using the laser irradiation and ablation parameters. The experiment determined the threshold for laser damage under pulses of 1030 nm wavelength and 224 fs duration; with a beam spot size of 9.6  $\mu$ m. The threshold energy density varied from 25.99 to 16.29 J/cm<sup>2</sup> with pulse numbers varying from 1 to 20,000. The threshold power density of incident radiation ranged from 11.6 to 7.3 GW/cm<sup>2</sup>.

### KEYWORDS

gallium oxide • laser • optical resistance • Liu's method • laser ablation • laser radiation

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## Introduction

Beta-gallium oxide ( $\beta$ -Ga<sub>2</sub>O<sub>3</sub>) has emerged as a leading ultra-wide bandgap (UWBG) semiconductor, with a direct bandgap of  $\sim$  4.8 eV, surpassing the performance limits of conventional materials like SiC and GaN [1–5]. Its exceptional properties including a high critical electric field (8 MV/cm<sup>-1</sup>), excellent thermal stability, and the availability of high-quality substrates make it ideal for next-generation power devices, deep-ultraviolet (UV) photodetectors, and radiation-hardened applications [6–9].

Despite significant progress in understanding the electrical [10–14] and structural properties [14–20] of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>, critical challenges still remain in optimizing its performance and reliability for practical applications. One of the key issues is the material's response to various forms of radiation and environmental stress, particularly in optoelectronic applications where the material may be subjected to intense optical irradiation.

The beta phase of gallium oxide belongs to the monoclinic system, spatial group C2/m [21] with lattice parameters  $a = 12.214$  Å,  $b = 3.0371$  Å,  $c = 5.7981$  Å,  $\beta = 103.83^\circ$ . The unit cell of the crystal has three different oxygen positions, designated as O(1), O(2) and O(3), and two gallium positions, Ga(1) and Ga(2). Nodes O(1) and O(3) have the smallest coordination number 3, which leads to weak interplanar connections in planes (100) and (001) [22,23]. Due to the peculiarities of the crystal lattice, gallium oxide is an anisotropic material. It has been experimentally shown that a number of characteristics



(for example, thermal conductivity or electron mobility) have better values along the [010] direction [22–24]. However, mechanical processing is difficult due to fragility and mechanical defects of the material in the plane (010). As a result, other processing methods for  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> are being studied, including laser scribing [25–28]. Yoo J.H. et al. [29] studied the optical resistance of bulk gallium oxide, but these samples were doped with Sn, which makes its own adjustments to the optical resistance when compared with undoped samples. The paper presents the results of determining the optical resistance of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> surface under the action of femtosecond laser pulses of the near-IR spectrum depending on the number of pulses and their repetition frequency from 10 to 201 kHz.

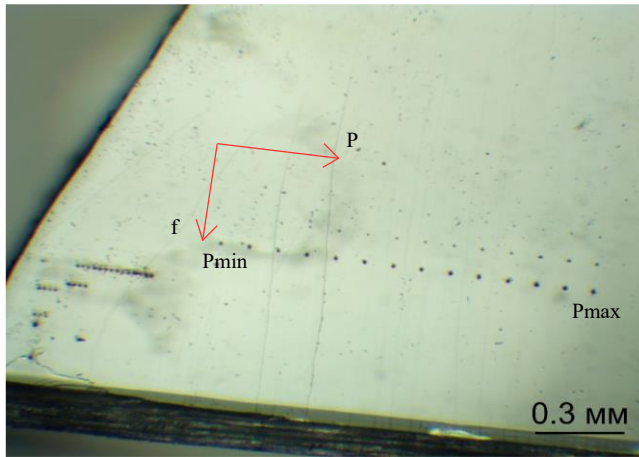
## Materials and Methods

The sample of bulk crystal for studying the optical resistance of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> was grown by Czochralski method using the NIKA-3 growth unit. The crystal growing process is described in detail in the paper [30]. The sample was prepared by method of chipping the bulk  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> crystal along the cleavage plane (100). The ablation of the material was performed on an experimental setup equipped with an ANTAUS-20W Yb-fiber femtosecond laser (Avesta) and a 3D coordinate moving system based on DDSM50 and MTS50M-Z8 translators (Thorlabs). For investigation the laser beam with Gaussian intensity profile, factor  $M^2 < 1.1$ , maximum average optical power  $P_{\max} = 17.5$  W at the pulse repetition frequency of 1 MHz, pulse duration of 224 fs, output beam diameter of 2.0 mm, and quasi-monochromatic wavelength 1030 nm, which transparent to  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>, was used [1]. In the experiment, the laser beam was focused using a 40X, NA 0.60 objective lens into a spot with a diameter of 9.6  $\mu$ m on the surface of the sample, while the latter was rigidly fixed on the coordinate table. Ablated areas were formed at the optical power in the range from 0.06 to 0.19  $P_{\max}$ , fixed exposure time of 0.1 s and different repetition pulse frequency: 201·10<sup>3</sup>, 2·10<sup>3</sup>, 200 and 10 Hz, respectively. As a result, the pulses number and time between its were varied as follows: 20,000 pulses every 5  $\mu$ s, 200 pulses every 0.5 ms, 20 pulses every 5 ms and single pulse. Gentec Solo PE-2 M (Lake Oswego) optical power meters equipped with UP19K-110F-H9 pyroelectric power detector (Lake Oswego) used for registration of incident laser power. The uncertainty of the power meter calibration was  $dP = \pm 2.5$  %.

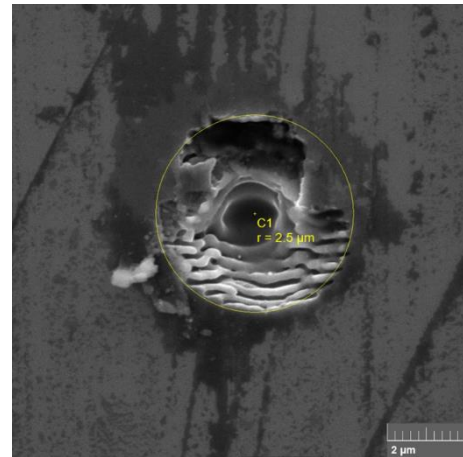
## Results and Discussion

To determine the optical resistance of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> sample, a series of experiments with ablated areas formation by different number pulses with a laser beam power in the range from 1.05 to 3.33 W. Figure 1 shows an optical microscope image of the sample after laser ablation. By analyzing the images obtained using a scanning electron microscope (Fig. 2), the diameters of the ablation crater were measured depending on the frequency and power of laser ablation.

It's observed from Table 1 that the value of the diameters of crater has a stepwise increase depending on a laser power. Thus, laser ablation can be quantitatively characterized by the crater diameter, which is necessary for calculating the optical resistance of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>.



**Fig. 1.** An optical microscope image of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> after laser ablation on a scale of 0.3 mm, with arrows indicating an increase in repetition rate ( $f$ ) and optical power of laser beam ( $P$ )



**Fig. 2.** An example of measuring the diameter of an ablation crater using a scanning electron microscope

**Table 1.** Diameter of ablated areas of sample surface in microns with error  $\delta D = \pm 0.1 \mu\text{m}$

Number of pulses	Average optical power $P$ , W													
	1.05	1.22	1.40	1.57	1.75	1.92	2.10	2.27	2.45	2.62	2.80	2.97	3.15	3.32
20,000	5.4	5.5	5.5	5.5	5.7	5.7	6.5	8.1	8.2	8.3	8.3	8.5	8.5	9.3
200	2.5	4.2	5.0	5.3	5.5	5.5	5.7	5.9	6.0	7.5	7.6	7.7	8.0	8.1
20	2.0	2.4	2.4	2.4	4.0	4.5	4.9	5.0	5.0	5.1	5.2	5.0	5.9	6.0
1	1.5	1.5	1.6	1.7	1.8	1.6	1.5	3.5	4.2	4.3	4.5	4.5	4.5	4.4

### Calculation of the optical resistance

The optical resistance of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> surface was determined by measuring the diameter of the craters  $D$  formed as a result of laser ablation at different pulse energies  $E$  using the Lui method [31]. Depending on the diameter of the wells squared from the energy of the impulses at which they were formed, a linear regression was given by the expression:

$$D^2(E) = 2\omega_0^2 \ln(E/E_{th}), \quad (1)$$

where  $\omega_0$  is the radius of the laser spot determined by the intensity drop on  $e^{-2}$ , which formed the crater. The energy in the pulse was calculated from the average power of incident radiation  $P$ , measured at the pulse repetition frequency  $f = 20$  kHz, as:

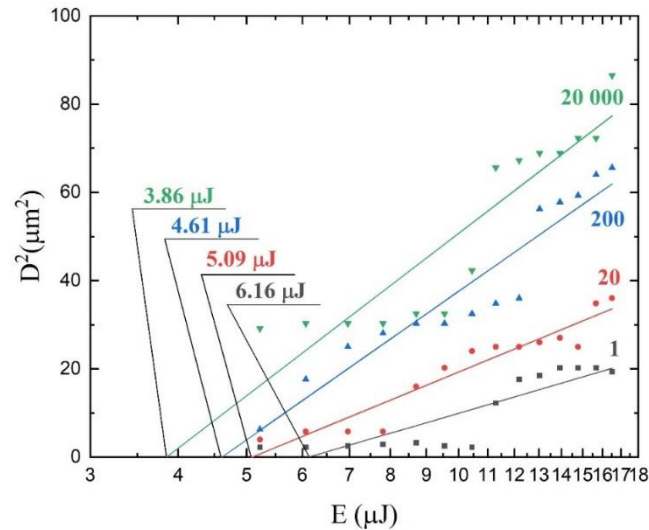
$$E = P/f. \quad (2)$$

According to Eq. (1), the graphical dependence  $D^2(E/E_{th})$  was plotted on a logarithmic scale along the abscissa axis. In this case, the point of intersection of the linear regression with the abscissa axis determined the pulse energy  $E_{th}$ , at which the formation of the well began, and the slope of the line corresponded to the radius of the laser spot:

$$\omega_0 = [-b/(2E_{th}) \cdot N_{ph}]^{0.5}, \quad (3)$$

where  $N_{ph}$  is the value of multiphoton absorption,  $b$  is the linear regression constant written as a function  $y = kx + b$ . This function was plotted based on experimental data by least squares approximation (Fig. 3). For all points, the exposure duration was 0.1 seconds. 20,000 pulses corresponded to a frequency of 201 kHz, 200 pulses corresponded to 2.01 kHz, 20 pulses corresponded to a frequency of 201 Hz and 1 pulse at 10 Hz.

The value of  $N_{ph}$  was determined as the ratio of the energy of the bandgap of the material  $E_g$  to the photon energy  $E_{ph}$  and characterized the cross-section of multiphoton absorption, i.e. the region of the Gaussian laser beam where the energy density provided  $N_{ph}$ -photon absorption of incident radiation. In our case, at  $E_g = 4$  eV and  $E_{ph} = 1.2$  eV (the energy of a photon with a wavelength of 1030 nm), the value of  $N_{ph} = 4$ .



**Fig. 3.** Dependence of the square of the sublimation diameter on the pulse energy of the different pulses in the irradiated region: 1, 20, 200 and 20,000 pulses

Optical resistance of the sample was characterized by the energy density at which surface ablation began, and was expressed as follows:

$$F_{th} = E_{th}/(\pi\omega_0^2). \quad (3)$$

The constants of the linear regression, as well as the values of the laser spot radius and threshold energy density of the material are shown in Table 2. From the data provided, it can be seen that the threshold energy of  $E_{th}$  was maximum when exposed to a single pulse, equal to 6.16  $\mu\text{J}$  and decreased almost twice – to 3.86  $\mu\text{J}$  – with an increase in the number of pulses to 20,000. The radius of the laser spot did not depend on the number of pulses and was supposed to be 7.7  $\mu\text{m}$  for all irradiation modes. An increase in the radius of the laser beam according to the computational data under the influence of 200 and 20,000 pulses indicated that the size of the ablative crater became larger than the laser spot due to spalling ablation. In this case, part of the material outside the irradiated zone was broken off and removed, thereby increasing the diameter of the ablation zone from 15.4 to 24.4  $\mu\text{m}$ .

**Table 2.** Linear regression constants, threshold pulse energy, beam radius, threshold energy and power densities for optical resistance

Number of pulses/ repetition rate	$b$	$k$	$E_{th}, \mu\text{J}$	$\omega_0, \mu\text{m}$	$F_{th}, \text{J}/\text{cm}^2$	$W_{th}, \text{W}/\text{cm}^2$
1 / 10 Hz	$-46.47 \pm 5.96$	$65.76 \pm 5.83$	6.16	7.77	$25.99 \pm 4.21$	$11.6 \cdot 10^9$
20 / 201 Hz	$-36.81 \pm 7.60$	$46.75 \pm 7.43$	5.09	7.61	$21.48 \pm 1.33$	$9.6 \cdot 10^9$
200 / 2,01 kHz	$-74.09 \pm 10.64$	$111.68 \pm 10.41$	4.61	11.34	$19.45 \pm 3.35$	$8.7 \cdot 10^9$
20,000 / 201 kHz	$-71.54 \pm 16.04$	$122.23 \pm 15.69$	3.86	12.18	$16.29 \pm 4.22$	$7.3 \cdot 10^9$

The measurement error was estimated considering linear regression, the accuracy of measuring the diameter of the wells and the incident radiation power, for which an expression of the following form was used:

$$dF = F_{th} \sqrt{\left(\frac{dk}{k}\right)^2 + \left(\frac{db}{b}\right)^2 + \frac{1}{14} \sum_{i=1}^{14} \left(\frac{\delta D_i}{D_i}\right)^2 + \delta P^2}. \quad (4)$$








As a result, firstly, the average measurement error ranged from 16.4 % when exposed to 1 pulse to 26 % after exposure to 20,000 pulses. Measurement error can be reduced by recording not one, but several series of wells under the same laser exposure regimes. Secondly, the minimum error under the same other irradiation conditions was for the effect of a single pulse. Thirdly, ablation of the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> surface under the influence of high-power near-infrared laser pulses occurred due to multiphoton absorption, which was random, unstable and contributed to a high measurement error. Reducing the wavelength to visible and near-UV radiation would also reduce measurement error.

From the above data of the threshold energy density  $F_{th}$ , it can be noted that its value decreased by 37 % with an increase in the number of pulses from 1 to 20,000 (Table 2). This result was typical for any materials in the transition from single-pulse to multi-pulse action. With each subsequent pulse, the fatigue of the material accumulated in the form of an increase in the concentration of local defects in the crystal lattice

## Conclusions

Experiments on the effects of laser irradiation were carried out on samples of bulk crystals of gallium oxide grown using the Czochralski method and prepared by chipping along the cleavage plane. During the analysis of the laser ablation geometric parameters and calculations, the optical resistance of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> was determined. The threshold energy density varied from 25.99 to 16.29 J/cm<sup>2</sup> with pulse numbers varying from 1 to 20,000 at a wavelength of 1030 nm and a pulse duration of 224 fs. The threshold power density of incident radiation ranged from 11.6 to 7.3 GW/cm<sup>2</sup>.

## CRedit authorship contribution statement

**Dmitrii I. Panov** : writing – review & editing, writing – original draft, conceptualization, data curation; **Nikita A. Balabanov**: writing – original draft, conceptualization, investigation; **Maxim M. Sergeev**: writing – review & editing, conceptualization, investigation; **Vladislav A. Spiridonov**  : writing – review & editing, investigation ; **Dmitrii A. Bauman**  : writing – review & editing; **Alexey E. Romanov**  : writing – review & editing, supervision

## Conflict of interest

The authors declare that they have no conflict of interest.

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