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Electrospun Nanofibers Based on Gallium Oxide: Fabrication and Characterization

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Abstract

Currently, nanofibrous semiconductor materials having large surface area and wide bandgap are in demand for modern technological processes, from fabrication of optoelectronic devices to photocatalytic facilities. Ga_2O_3 is the most suitable semiconductor for such materials due to its unique properties, wide bandgap equal to ~4.8 eV, and high acid resistance. In this study, Ga_2O_3-nanofibers were fabricated by electrospinning technique from the polymer spinning solutions based on polyvinylpyrrolidone. The fabrication procedure consists of two stages: electrospinning of nanofibers loaded with the gallium oxide precursor and annealing of nanofibers obtained for polymer removal and Ga_2O_3 formation. Influence of annealing temperature on the fiber morphology and its optic-electronic properties were demonstrated. Results obtained provide experimental basis for further fabrication of metal-oxide nanofibers, including doped ones, for high effective devices.

Keywords: Electrospinning; Nanofibers; Gallium oxide; Size distribution; Bandgap

1. INTRODUCTION

Photocatalytic materials are currently widely utilized in various fields, including water and air purification, solar energy, self-cleaning surfaces, temperature sensors and biomedical devices [1–4]. Nanomaterials in this direction are particularly promising due to their large specific surface area and porous structure, which significantly enhance the efficiency of photocatalytic processes [5–7].

Among semiconductors, gallium oxide (β-Ga_2O_3) has attracted significant interest. This material exhibits an ultra-wide bandgap (~4.8 eV), high thermal stability, and chemical inertness, particularly resistance to corrosion in aggressive environments. These unique properties make β-Ga_2O_3 a highly promising photocatalytic material [8–9].

Nanofibers of β-Ga_2O_3 fabricated via electrospinning offer numerous advantages. Their high porosity and large surface-to-volume ratio facilitate improved contact with reactants and efficient light absorption. Unlike nanoparticle synthesis, the electrospinning method enables the production of nanofibers without the need for additional substrates, simplifying the technological process and promoting the formation of structures with uniform morphology.

A recently published review by Snetkov et al. [10] systematizes advancements in the fabrication of Ga_2O_3 nanofibers via electrospinning. The reviewed studies analyze various electrospinning parameters, compositions of precursor solutions, and the physicochemical properties of the resulting fibers, establishing a foundation for further research in this field.

The aim of the present study is to fabricate β-Ga_2O_3 nanofibers with controlled morphology using the electrospinning method and to comprehensively investigate their properties.

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2. MATERIALS AND METHODS

To prepare nanofibers, high-molecular-weight polyvinylpyrrolidone (PVP, K-90, molecular weight 1.5 MDa, Ashland Global Holdings Inc., USA) was used as the polymer matrix, and gallium nitrate octahydrate ($Ga(NO_3)_3 \cdot 8H_2O$, 99.9%, Chemcraft Ltd., Russia) served as the precursor for gallium oxide. The polymer concentration was varied from 5 to 10 wt.%, while the precursor concentration varied from 0.5 wt.% to 6 wt.%. The solvent consisted of an equal-volume mixture of distilled water and ethanol (EtOH, 95.0%, Vekton Ltd., Russia). All solutions were stirred at room temperature until complete dissolution of the components was achieved.

Experiments on the fabrication of gallium oxide nanofibers via electrospinning were conducted under controlled laboratory conditions. The temperature in the laboratory setup was maintained at 25 ± 1 °C, with a relative humidity of 30 ± 1%.

The electrospinning process lasted 40 minutes, followed by additional drying of the fibers in the laboratory setup for 15 minutes. After fiber formation, the samples were dried at room temperature for 48 hours to remove residual solvent traces.

Subsequent annealing was performed at three temperature regimes: 600, 750, and 900 °C, with a heating rate of 5 °C/min. After reaching the target temperature, the samples were held for 240 minutes, followed by cooling in a switched-off furnace. The thermal treatment facilitated the removal of the polymer and the formation of gallium oxide from the precursor.

The morphology of the nanofibers was investigated using scanning electron microscopy (SEM) with a MIRA3 TESCAN microscope without prior sputter coating and measured using ImageJ software. For each precursor concentration, over 300 measurements were conducted. Based on the obtained data, histograms of diameter distributions were constructed using the NumPy and Matplotlib libraries in Python, and statistical analysis was performed to calculate mean values and standard deviations.

A semi-quantitative assessment of elemental content and distribution were performed by energy-dispersive spectroscopy (EDS). Analysis was conducted using a MIRA3 TESCAN scanning electron microscope equipped with an EDS detector.

Given that β-Ga_2O_3 is a direct bandgap semiconductor, the optical bandgap was determined using the Tauc-plot method for direct transitions [11]. Diffuse reflectance spectra were obtained using a Lambda 1050 spectrophotometer (PerkinElmer, USA) in the wavelength range of 200–450 nm. Measurements were conducted in diffuse reflectance mode using an integrating sphere, which ensured the averaging of scattered light from all directions.

3. RESULTS AND DISCUSSION

One of the key challenges in the field is the fabrication of nanofibers with well-defined morphological characteristics. In addressing this issue, we accomplished two main objectives: we optimized the processing parameters for the electrospinning of gallium oxide nanofibers and investigated their key morphological characteristics.

To identify the optimal electrospinning conditions, we systematically varied key processing parameters. As a result, the following combination was found to produce gallium oxide nanofibers with the desired morphology: a high-voltage power supply was used to apply a potential of 25 kV, generating an electric field between the needle and the collector; the precursor solution was delivered at a controlled flow rate of 0.03 mL/min using a syringe pump; and the distance between the needle and the collector was set at 21 cm. The polymer concentration was equal to 7 wt.%, while the precursor concentration varied from 0.5 wt.% to 6 wt.%.

The following solutions (20 mL each) were prepared:
1) PVP + H_2O/EtOH;
2) PVP + H_2O/EtOH + Ga (0.5 wt.%);
3) PVP + H_2O/EtOH + Ga (1 wt.%);
4) PVP + H_2O/EtOH + Ga (1.5 wt.%);
5) PVP + H_2O/EtOH + Ga (2 wt.%);
6) PVP + H_2O/EtOH + Ga (4 wt.%);
7) PVP + H_2O/EtOH + Ga (6 wt.%).

3.1. Scanning electron microscopy

SEM images of morphology are presented in Fig. 1. Diameter distribution histograms are shown in Figs. 2–7.

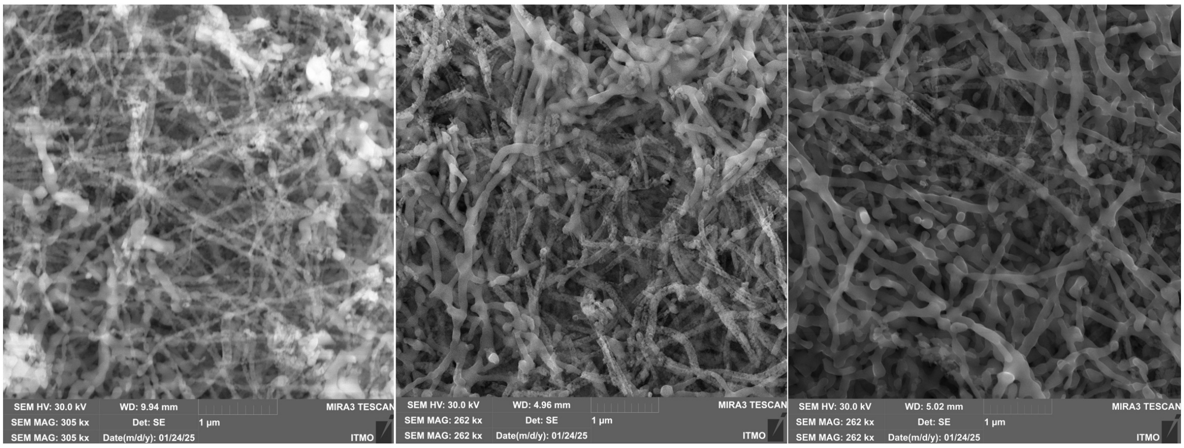
A comparison of the diameters of annealed gallium oxide nanofibers with blank polymer nanofibers is presented in Fig. 8.

A comparison of the diameters of annealed gallium oxide nanofibers is shown in Fig. 9. The dashed line indicates the mean diameter of the nanofibers.

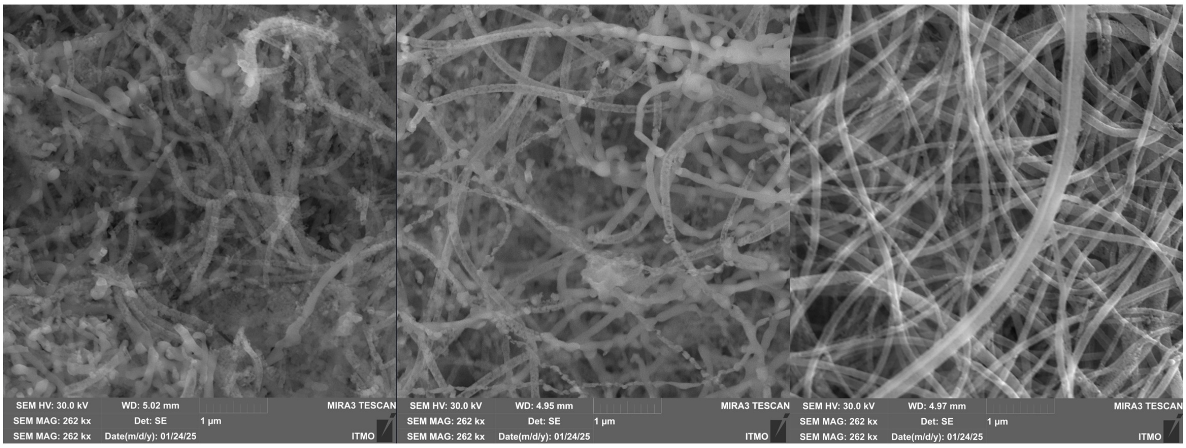
Results are summarized in Table 1.

At low Ga concentrations, smaller average fiber diameters are observed, with distribution peaks in the range of 105–140 nm. These distributions are narrower, indicating a higher degree of size uniformity. As the Ga concentration increases, the diameter distribution shifts toward larger values. For higher concentrations, such as 4%, the peak shifts further to the right, reaching the 120–140 nm range. By contrast, Y. Zhang et al. [12] obtained wider range of diameters (from 158 to 772 nm) using H_2O/EtOH = 3/1 ratio. Thus, it is supposed that variation in EtOH concentration leads to changes in nanofibers diameters' range.

Notably, the 6% Ga sample deviates from the overall trend of increasing diameter with increasing Ga content.



(a) (b) (c)



(d) (e) (f)

Fig. 1. SEM images of obtained nanofibers: (a) 0.5% Ga, (b) 1% Ga, (c) 1.5% Ga, (d) 2% Ga, (e) 4% Ga, (f) 6% Ga.

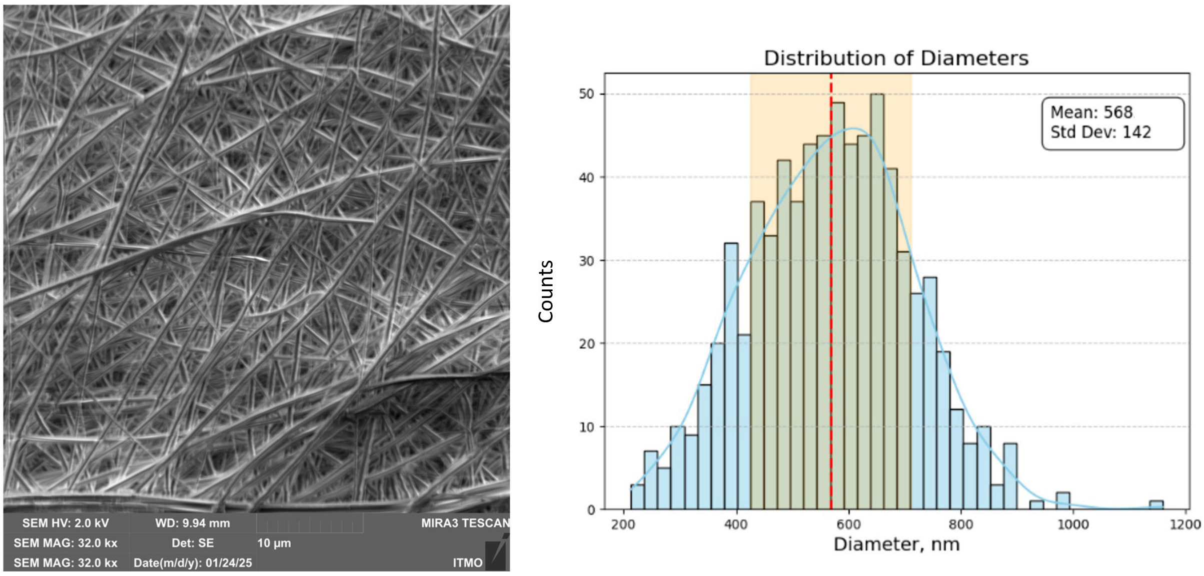


Fig. 2. SEM image and diameter distribution histogram of blank nanofibers based on PVP. The average diameter is 568 ± 142 nm (mean ± standard deviation).

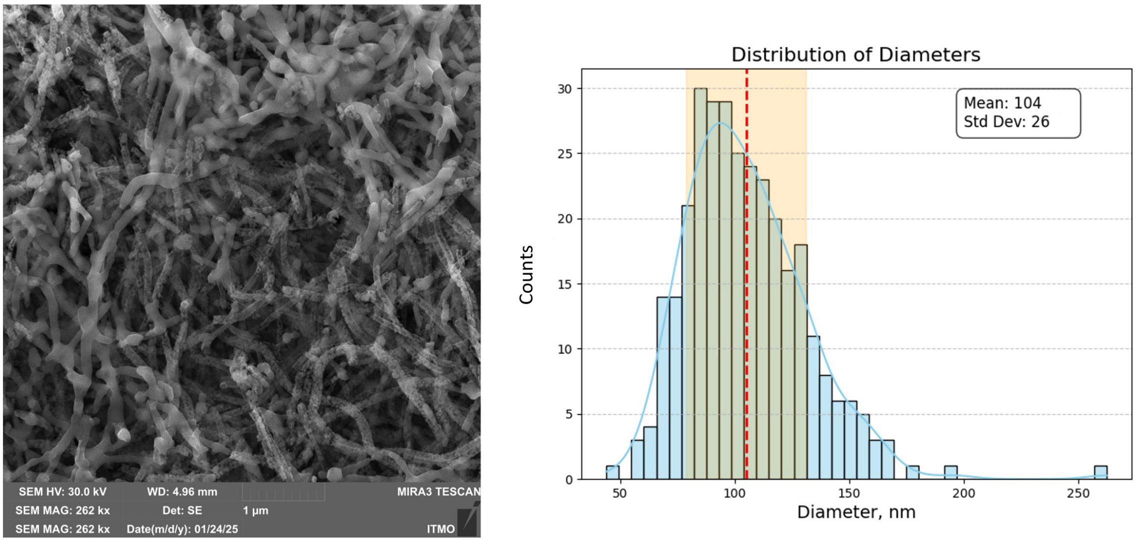


Fig. 3. SEM image and diameter distribution histogram of annealed nanofibers electrospun from polymer solution with 1 wt.% Ga. The average diameter is 104 ± 26 nm.

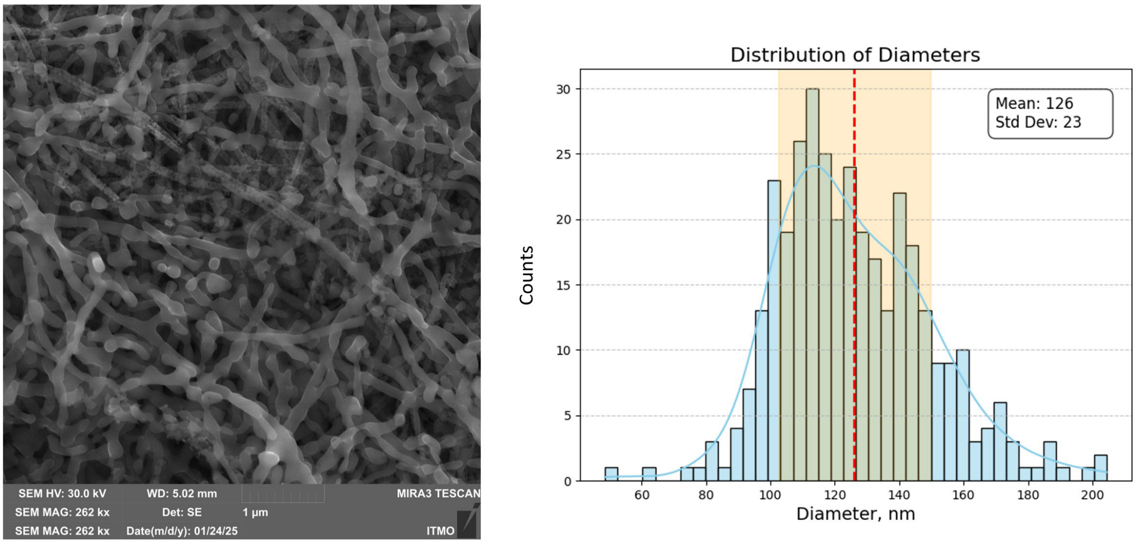


Fig. 4. SEM image and diameter distribution histogram of annealed nanofibers electrospun from polymer solution with 1.5 wt.% Ga. The average diameter is 126 ± 23 nm.

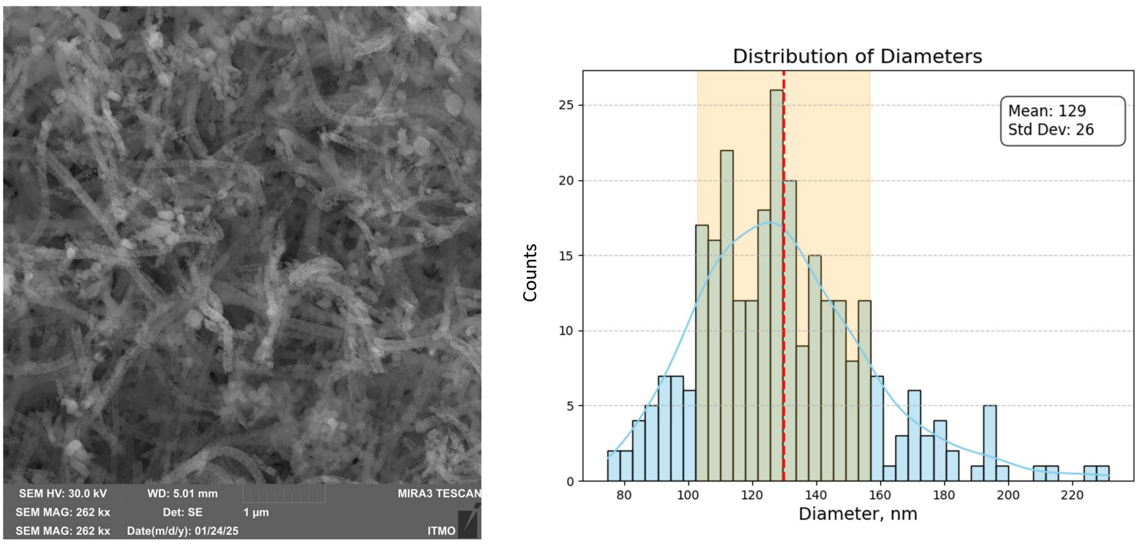


Fig. 5. SEM image and diameter distribution histogram of annealed nanofibers electrospun from polymer solution with 2 wt.% Ga. The average diameter is 129 ± 26 nm.

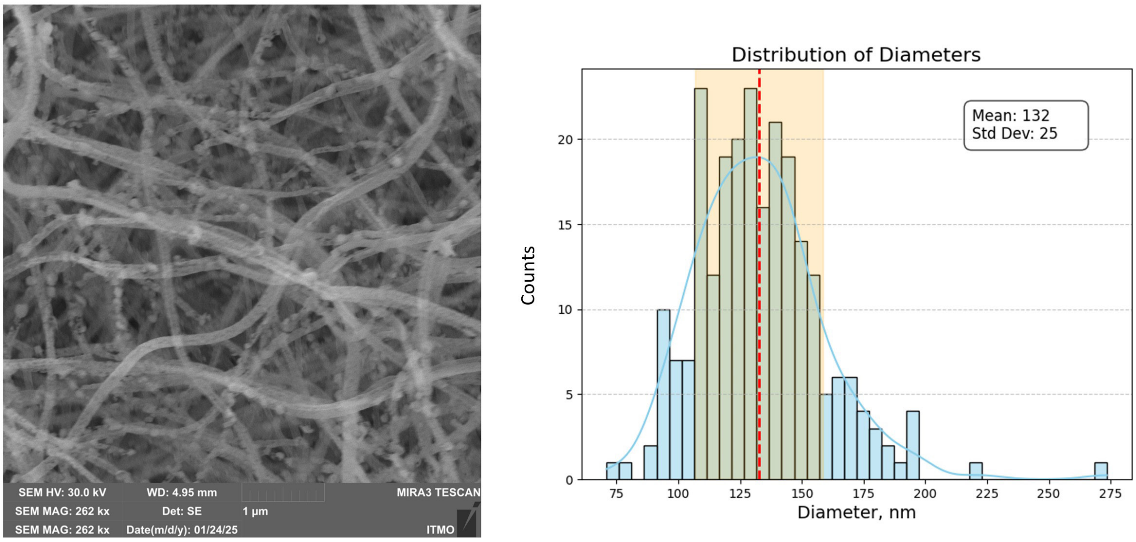


Fig. 6. SEM image and diameter distribution histogram of annealed nanofibers electrospun from polymer solution with 4 wt.% Ga. The average diameter is 132 ± 25 nm.

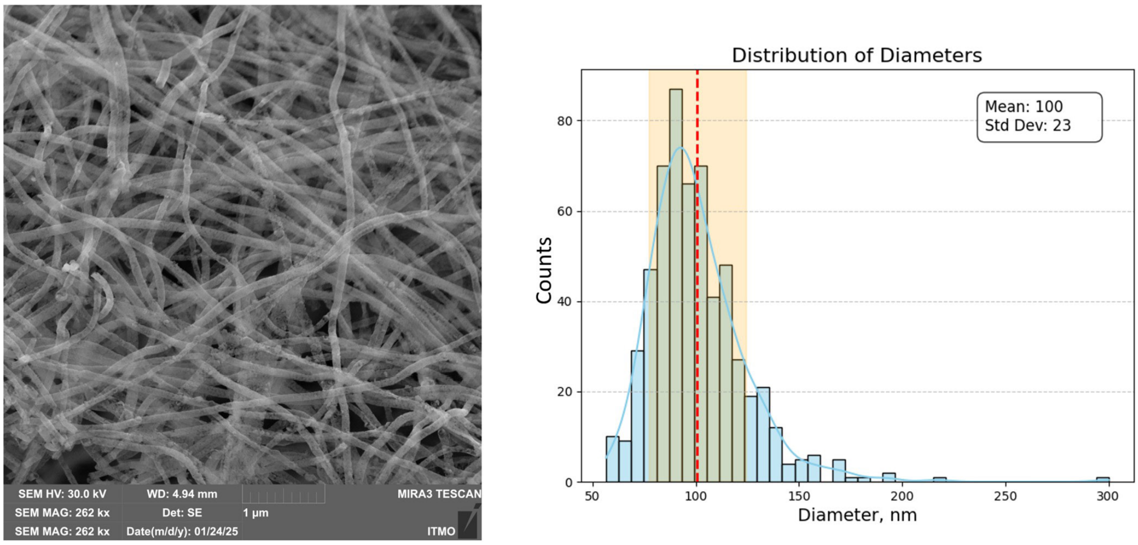


Fig. 7. SEM image and diameter distribution histogram of annealed nanofibers electrospun from polymer solution with 6 wt.% Ga. The average diameter is 100 ± 23 nm.

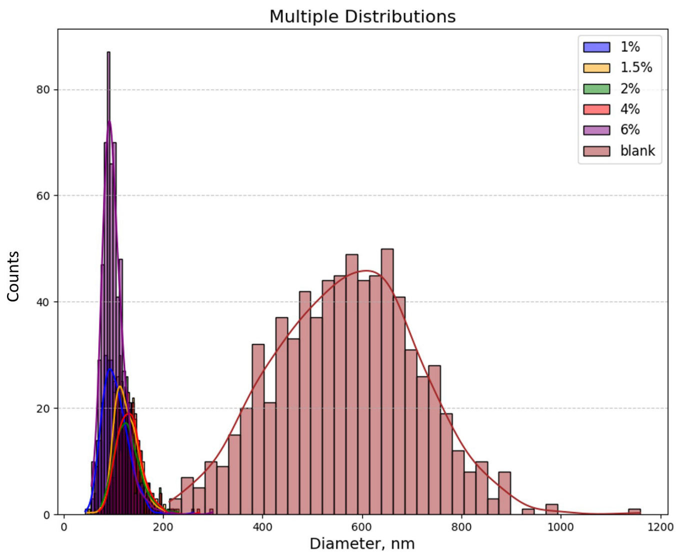


Fig. 8. A comparison of the diameters of annealed gallium oxide nanofibers with blank polymer nanofibers.

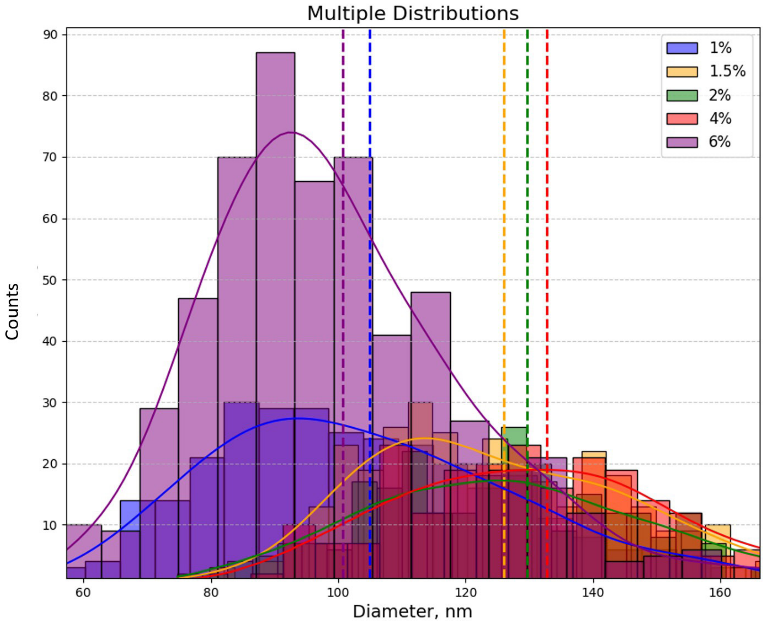


Fig. 9. A comparison of the diameters of annealed gallium oxide nanofibers.

Table 1. A comparison of the diameters (given in nm) of annealed gallium oxide nanofibers with untreated nanofibers.

Ga content	Annealed			Untreated
	600 °C	750 °C	900 °C	
0.5%	74 ± 18	75 ± 15	–	316 ± 71
1%	97 ± 20	97 ± 16	104 ± 26	289 ± 65
1.5%	107 ± 21	114 ± 18	126 ± 23	300 ± 69
2%	118 ± 25	114 ± 25	129 ± 26	270 ± 64
4%	121 ± 26	117 ± 20	132 ± 25	282 ± 69
6%	120 ± 29	165 ± 52	100 ± 23	274 ± 64

Further investigation is planned to determine whether this result represents a statistical outlier.

Thus, low Ga concentrations promote the formation of thinner fibers with narrow diameter distributions, whereas higher concentrations lead to an increase in both fiber diameter and dispersion. This tendency is in agreement with the results obtained earlier [13].

3.2. Energy-dispersive spectroscopy

The sample composition primarily consists of 60.0 at.% of oxygen (O) and 38.9 at.% gallium (Ga). The theoretical stoichiometric composition of gallium oxide suggests an atomic ratio of 2Ga:3O, equivalent to 40 at.% Ga and 60 at.% O. Thus, the obtained values are very close to the theoretical ones. The slight reduction in gallium content by approximately 1.1 at.% can be attributed to methodological errors.

EDS mapping (Fig. 10) revealed a uniform distribution of the main elements across the surface, indicating the homogeneity of the obtained nanofibers. The results of the EDS analysis confirm the successful synthesis of gallium oxide nanofibers with an elemental composition closely approximating the stoichiometric ratio.

3.3. UV-Vis diffuse reflectance spectroscopy

The original reflection spectra of Ga_2O_3 nanofibers obtained, presented in Fig. 11, were converted to the Kubelka-Munk function, $F(R)$, which provides a quantity proportional to the absorption coefficient. $[h\nu F(R)]^2$ was plotted against the photon energy $h\nu$, and the bandgap energy E_g was obtained, as shown in Fig. 12.

The calculated bandgap values for samples annealed at different temperatures are summarized in Table 2.

The results of the band gap measurements indicate that at temperatures of 750 °C and 900 °C, the β-phase

Table 2. Calculated bandgap values.

Annealing temperature, °C	Bandgap, eV
600	4.77
750	4.84
900	4.86

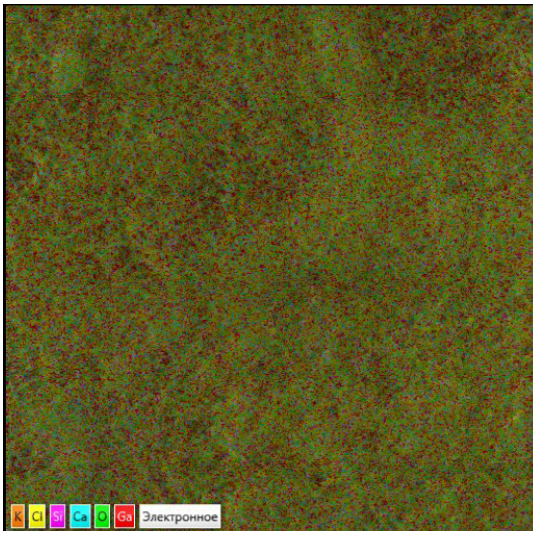


Fig. 10. EDS mapping of obtained Ga_2O_3 nanofibers.

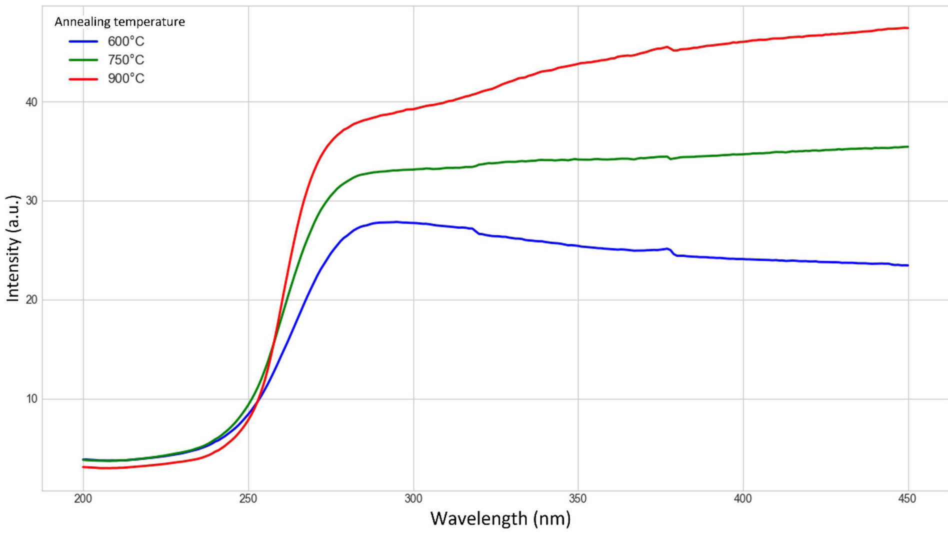


Fig. 11. Reflection spectra of obtained Ga_2O_3 nanofibers.

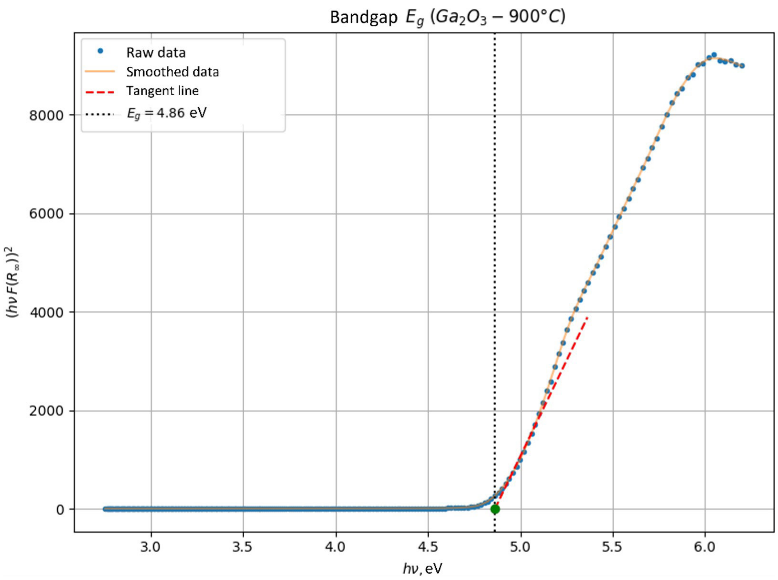


Fig. 12. The bandgap energy E_g of obtained Ga_2O_3 nanofibers.

of gallium oxide is formed, which is consistent with the previously obtained data [14].

4. CONCLUSIONS

The development of fabrication methods for Ga_2O_3 nanofibers is of significant importance due to the unique properties of gallium oxide. In this study, we present a controlled fabrication method for Ga_2O_3 nanofibers and investigate their structural and optical properties. The morphology of the nanofibers was examined using scanning electron microscopy (SEM), enabling the analysis of the effect of precursor concentration on fiber diameter. Energy-dispersive spectroscopy (EDS) confirmed the successful synthesis of Ga_2O_3 nanofibers with an elemental composition close to stoichiometry. UV-Vis spectrophotometry was employed to determine the optical band gap of samples annealed at various temperatures. The results demonstrate that electrospinning is an effective method for producing Ga_2O_3 nanofibers with controlled morphology. The identified correlation between precursor concentration and fiber diameter provides valuable insights for optimizing the fabrication process. The findings of this study contribute to the ongoing development of nanomaterials and their potential applications in advanced technological fields.

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Электроформованные нановолокна на основе оксида галлия: получение и свойства

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Аннотация. В настоящее время нановолоконные полупроводниковые материалы, обладающие большой удельной поверхностью и широкой запрещённой зоной, востребованы в современных технологических процессах: от производства оптоэлектронных устройств до фотокаталитических систем. Оксид галлия (Ga_2O_3) является одним из наиболее подходящих полупроводников для таких материалов благодаря своим уникальным свойствам, широкой запрещённой зоне (~4,8 эВ) и устойчивости к кислотам. В данном исследовании нановолокна Ga_2O_3 были получены методом электроформования из полимерных растворов на основе поливинилпирролидона. Процесс получения включал два этапа: электроформование нановолокон, содержащих прекурсор оксида галлия, и последующий отжиг для удаления полимера и формирования оксида галлия. Показано влияние температуры отжига на морфологию волокон и их оптоэлектронные свойства. Полученные результаты служат экспериментальной основой для дальнейшей разработки нановолокон на основе оксидов металлов для создания высокоэффективных устройств.

Ключевые слова: электроформование; нановолокна; оксид галлия; распределение по диаметрам; ширина запрещенной зоны