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THE EFFECT OF PREPARATION CONDITIONS ON THE ELECTRICAL CONDUCTIVITY OF THIN FILMS OF $(Y_{0.06}Ga_{0.94})_2O_3$

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ABSTRACT

Background. β - Ga_2O_3 gallium oxide is a promising wide-bandgap semiconductor widely used in optoelectronic and sensing applications. The electrical conductivity of thin films strongly depends on their structural quality, defect states, and post-deposition treatment conditions. In polycrystalline films, grain boundaries and defect complexes significantly affect charge transport mechanisms. The objective of this study is to investigate the influence of annealing atmospheres on the structural, morphological, and electrical properties of $(Y_{0.06}Ga_{0.94})_2O_3$ thin films.

Materials and Methods. Thin films of $(Y_{0.06}Ga_{0.94})_2O_3$ with thicknesses of 0.3–1.0 μm were deposited by RF ion-plasma sputtering onto fused quartz substrates. Post-deposition annealing was carried out in oxygen and argon atmospheres at 1000–1100 °C, and in hydrogen at 600–650 °C. Structural properties were analyzed using X-ray diffraction, while surface morphology was examined by atomic force microscopy. Electrical conductivity was measured in the temperature range of 300–450 K, and activation energies were determined from temperature-dependent conductivity data.

Results and Discussion. X-ray analysis confirmed the formation of films in the monoclinic β - Ga_2O_3 phase, with enhanced crystallinity and preferred orientation after annealing in oxygen. It has been established that freshly deposited films have a high resistivity ($\rho > 10^{11} \Omega \cdot cm$), which decreases with increasing temperature and after annealing. Oxygen annealing resulted in activation energy of ~0.87 eV, while argon annealing produced higher values (~1.38 eV in 300–400 K range), indicating deeper donor levels associated with oxygen vacancies. Hydrogen annealing significantly reduced resistivity (~ $10^8 \Omega \cdot cm$) and activation energy (~0.40 eV), attributed to shallow donor states.

Conclusion. The electrical conductivity of $(Y_{0.06}Ga_{0.94})_2O_3$ thin films is governed by defect-related donor levels formed during annealing. Oxygen and argon atmospheres promote deep donor states, while hydrogen enhances shallow donor formation, leading to improved electrical conductivity.

Keywords: thin films, gallium oxide, crystalline structure, impurities, electrical conductivity



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INTRODUCTION

Gallium oxide (Ga_2O_3), particularly its β -phase, is one of the most promising wide-bandgap semiconductors due to its large bandgap ($\sim 4.8\text{--}4.9$ eV), high electrical resistance, and stability, which ensures its widespread use in power electronics, UV photodetectors, and sensor systems [1–4]. An important feature of this material is the high sensitivity of its electrophysical and optical properties to the defect structure and doping [5–7].

One effective way to control the properties of Ga_2O_3 is through isovalent and heterovalent doping, particularly with rare-earth elements. The introduction of yttrium ions into the Ga_2O_3 crystal lattice leads to the formation of solid solutions of the $(\text{Y}_x\text{Ga}_{1-x})_2\text{O}_3$ type, which are characterized by altered structural, electronic, and luminescent properties [8]. In particular, the substitution of Ga^{3+} with larger Y^{3+} ions induces local lattice deformations that affect the energy spectrum of defect states and charge transport.

For thin films $(\text{Y}_{0.06}\text{Ga}_{0.94})_2\text{O}_3$, as well as for pure $\beta\text{-Ga}_2\text{O}_3$, electrical conductivity is largely determined by the defect subsystem, specifically oxygen vacancies and interstitial gallium atoms, which form donor levels in the bandgap [5, 9]. At the same time, yttrium doping can affect the concentration and stability of such defects by altering the energy parameters of the donor centers.

The conditions under which thin films are produced and subsequently heat-treated play a key role in determining their properties. It is known that annealing in various gas atmospheres (oxygen, inert gases, hydrogen) significantly affects the defect structure of the material, altering the concentration of oxygen vacancies and, consequently, electrical conductivity [10, 11]. In particular, reducing environments can promote the formation of shallow donor levels, while oxidizing environments can reduce their concentration.

In addition, the surface morphology and crystal structure of thin films, which depend on the deposition and annealing conditions, also significantly influence charge carrier transport. In polycrystalline films, grain boundaries create energy barriers and localized states that determine the mechanisms of electrical conductivity [12, 13].

Thus, investigating the effects of yttrium doping and heat treatment conditions on the structural, morphological, and electrical properties of thin films of $(\text{Y}_{0.06}\text{Ga}_{0.94})_2\text{O}_3$ is a pressing issue in modern semiconductor physics and materials science. Such studies allow not only for a deeper understanding of the nature of defect states but also for the optimization of material parameters for practical applications.

MATERIALS AND METHODS

Thin films of $(\text{Y}_{0.06}\text{Ga}_{0.94})_2\text{O}_3$ with thicknesses of $0.3\text{--}1.0$ μm were deposited by radio-frequency ion-plasma sputtering onto fused quartz ($\gamma\text{-SiO}_2$) substrates. After film deposition, the films were heat-treated in various gas atmospheres: in oxygen or argon at $1000\text{--}1100$ $^\circ\text{C}$, and reduced in hydrogen at $600\text{--}650$ $^\circ\text{C}$.

The results of X-ray diffraction analysis indicate the formation of a polycrystalline structure in the films, the characteristics of which vary depending on the chemical composition of the atmosphere and the heat treatment conditions. Typical diffractograms of the studied samples are shown in **Fig. 1**. Analysis of the obtained data showed that the crystalline structure of the films corresponds to the monoclinic phase of $\beta\text{-Ga}_2\text{O}_3$.

Analysis of the X-ray diffraction spectra of $(\text{Y}_{0.06}\text{Ga}_{0.94})_2\text{O}_3$ thin films following heat treatment in various atmospheres indicates that annealing conditions have a significant effect on their crystal structure and degree of order.

The results obtained show that for $(\text{Y}_{0.06}\text{Ga}_{0.94})_2\text{O}_3$ thin films, the nature of the crystallographic orientation significantly depends on the heat treatment conditions. After annealing in an oxygen atmosphere, a predominant orientation in the (110), (002), (111), and (512) planes is observed, with the reflection from the (110) plane being the most intense.

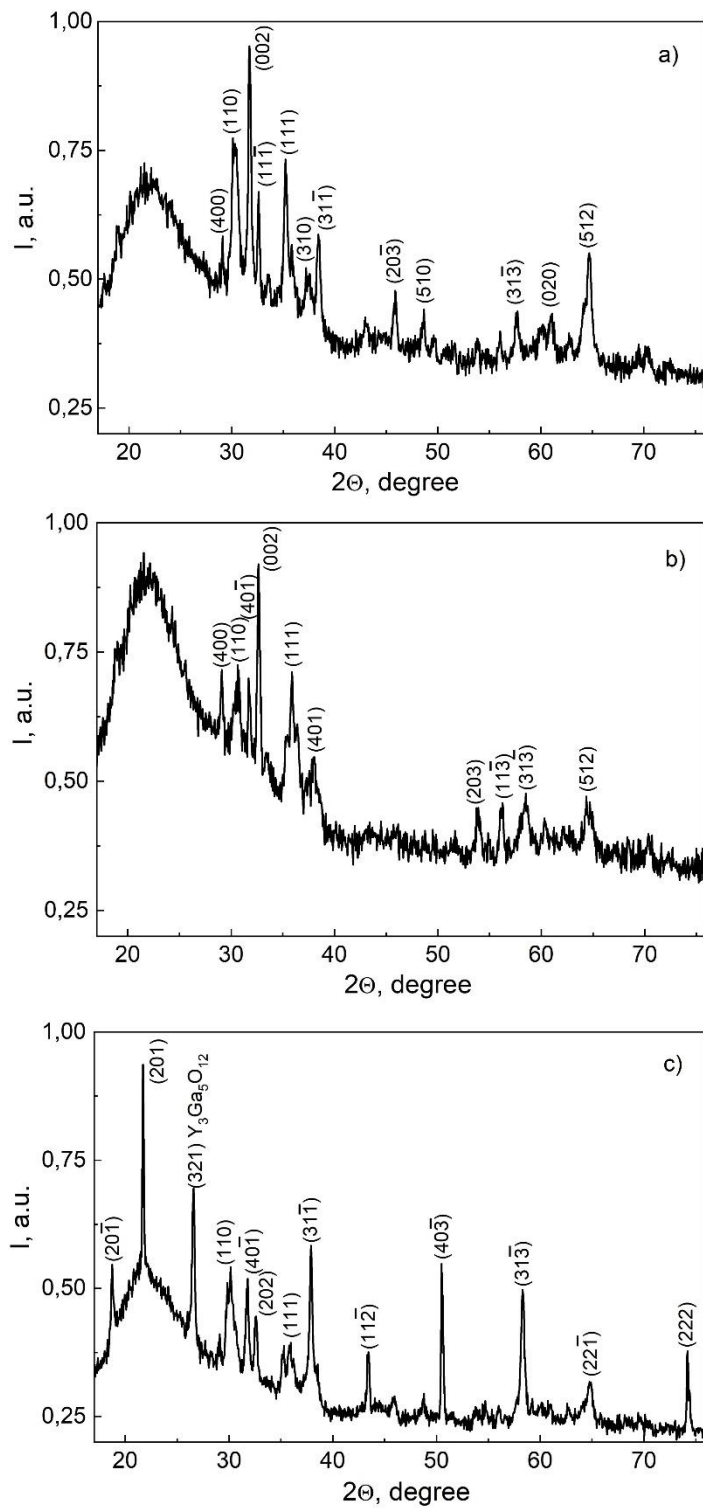


Fig. 1. Diffractograms (under $CuK\alpha$ irradiation) of thin films of $(Y_{0.06}Ga_{0.94})_2O_3$ obtained by RF ion-plasma sputtering, after heat treatment in an atmosphere of oxygen (a), argon (b), and hydrogen (c).

In the case of annealing in an argon atmosphere, the (002) and (111) planes become dominant, while the contribution of orientations associated with the (110) and (512) planes decreases. For films annealed in water, a redistribution of reflection peaks is observed, and the orientation of such films predominates in the (201), $(31\bar{1})$, $(40\bar{3})$, and $(31\bar{3})$ planes.

The elemental composition of the films was determined using an OXFORD INCA Energy 350 energy-dispersive spectrometer. Analysis conducted at several points on the surface confirmed that the experimentally obtained composition ratio corresponded to $(Y_{0.06}Ga_{0.94})_2O_3$.

Conductance measurements in the temperature range of 300–450 K were performed using an automated setup. A voltage in the range of 10–100 V was applied to two point contacts with a diameter of 1 mm, spaced 1 mm apart. When studying the electrical conductivity of thin $(Y_{0.06}Ga_{0.94})_2O_3$ films, it is fundamentally important to use ohmic contacts that do not create rectifying barriers at the interface. Such contacts are formed by materials that ensure effective electron injection into the film under forward bias and are characterized by a work function of approximately 4.5 eV.

In this study, polycrystalline carbon (Aquadag) was used as the contact material, as it meets the specified requirements. It is widely used as a conductive coating and material for forming ohmic contacts in studies of semiconductor and high-resistance materials, in particular oxide, carbide, and dielectric systems [5, 14, 15].

RESULTS AND DISCUSSION

To modify the electrical conductivity properties of $(Y_{0.06}Ga_{0.94})_2O_3$ thin-film phosphors, they were heat-treated in various gas atmospheres: oxygen, argon, and hydrogen. For the samples under study, the temperature dependence of electrical conductivity was determined, based on which the thermal activation energy of conductivity was calculated.

It has been established that after film deposition, $(Y_{0.06}Ga_{0.94})_2O_3$ films exhibit high specific resistance ($\rho > 10^{11} \Omega \cdot \text{cm}$) and low thermal activation energy, which is approximately 0.25 eV. Heat treatment in oxygen and argon atmospheres has virtually no effect on the specific resistance at room temperature (295 K). However, as the temperature rises to 450 K, a significant decrease in specific resistance is observed, down to values of the order of $10^8 \Omega \cdot \text{cm}$.

For films annealed in oxygen, the thermal activation energy for electrical conductivity is approximately 0.87 eV. In the case of annealing in argon, two temperature regions with different activation energy values were identified: in the 300–400 K range, it is about 1.38 eV, while at temperatures of 400–450 K, it decreases to approximately 0.40 eV (**Fig. 2**).

To further reduce the electrical resistance of the films, they were annealed in a reducing hydrogen atmosphere at a temperature of 650 °C. This treatment leads to a significant reduction in the specific resistance to a level of about $10^8 \Omega \cdot \text{cm}$, as well as to a decrease in the thermal activation energy of electrical conductivity to 0.25 eV (**Fig. 2**).

The characteristic values of the thermal activation energy of electrical conductivity for the studied films $(Y_{0.06}Ga_{0.94})_2O_3$ are given in **Table 1**.

To analyze the obtained dependencies, we will use the results of a study on electrical conductivity in unactivated $\beta\text{-Ga}_2\text{O}_3$. According to the results presented in the review [16], the gallium oxide $\beta\text{-Ga}_2\text{O}_3$ can exhibit both dielectric and semiconductor properties. Such changes are caused by variations in the synthesis of the samples. In particular, the

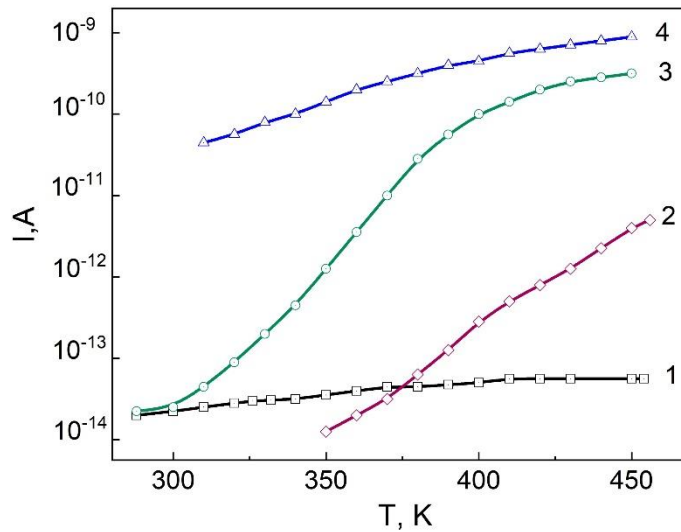


Fig. 2. Temperature dependence of the electrical conductivity of thin films of $(Y_{0.06}Ga_{0.94})_2O_3$ immediately after deposition (1) and after annealing in oxygen (2), argon (3), and hydrogen (4).

presence of oxygen vacancies and excess gallium atoms in Ga_2O_3 leads to the formation of donors and, consequently, n-type conductivity. Based on the results of [16, 17], it is evident that the conductivity of $\beta-Ga_2O_3$ crystals varies from 10^{-9} to $38 \Omega^{-1}\cdot cm^{-1}$ and is determined by the growth atmosphere. At the same time, according to [18], in thin-film samples of $\beta-Ga_2O_3$, the resistivity $\rho \approx 6 \times 10^{13} \Omega \cdot cm$.

The nature of electrical conductivity in thin films is more complex than in single-crystal materials. This is because films often have an imperfect structure—they may be amorphous or polycrystalline, and may also contain inclusions of other phases. An additional complicating factor is the presence of grain boundaries (GBs), which significantly affect the material's electrophysical properties.

In polycrystalline films, charge carriers move freely within the grains, but encounter energy barriers at their boundaries. Grain boundaries in oxide and semiconductor films act as energetically active regions where localized states form in the band gap. As a result, electrical conductivity is determined not only by the properties of the material itself but also by the energy levels associated with the GBs.

Table 1. Thermal activation energy for electrical conductivity in thin films of $(Y_{0.06}Ga_{0.94})_2O_3$

Thin film	Annealing atmosphere	Thermal activation energy of electrical conductivity, eV
$(Y_{0.06}Ga_{0.94})_2O_3$	Oxygen	0.87
$(Y_{0.06}Ga_{0.94})_2O_3$	Argon	1.38 (300-400 K) 0.40 (400-450 K)
$(Y_{0.06}Ga_{0.94})_2O_3$	Hydrogen	0.25

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Such centers can act as traps for electrons and holes, influencing recombination processes and charge carrier transport. Carrier trapping can occur both within grains and at grain boundaries, leading to a decrease in their mobility. If thermal energy is insufficient to release carriers from traps into the conduction band, a hopping mechanism of charge transport is realized. In this case, electrical conductivity increases with rising temperature and trap concentration.

At the same time, the precise determination of charge transport mechanisms requires a comprehensive analysis that includes not only the study of the temperature dependence of electrical conductivity but also additional experimental methods.

Annealing of films in a reducing hydrogen atmosphere is accompanied by the creation of a high concentration of oxygen vacancies and excess gallium atoms [19–22]. Defects of both types act as donors and lead to the emergence of n-type conductivity [15, 19]. As a result of such annealing, an increase in the conductivity of the studied films is observed. Two different types of defects in gallium oxide can possess donor properties—interstitial gallium atoms or vacancies in the oxygen sublattice [23–25].

Depending on the predominant type of defect formation reaction, different values of the parameter n appear in the following equation relating specific conductivity and oxygen partial pressure [26–28].

$$\sigma = kP_{O_2}^{-\frac{1}{n}} \quad (1)$$

Studies of the dependence of the conductivity of $(Y_{0.06}Ga_{0.94})_2O_3$ thin films on the partial pressure of oxygen P_{O_2} , measured at various temperatures, show that, depending on the degree of reduction, donor defects created by oxygen vacancies predominate in higher-resistance samples, while in more reduced samples, inter-site gallium ions predominate. Based on the results obtained, it can be assumed that in high-resistance $(Y_{0.06}Ga_{0.94})_2O_3$ thin films, oxygen vacancies form deep donor levels with activation energies of approximately 0.87 eV for samples annealed in oxygen, and approximately 1.38 eV in the temperature range of 300–400 K for films annealed in an argon atmosphere. The electrical conductivity of such films is determined by the thermal release of electrons from these deep levels.

The increase in the depth of donor levels in films annealed in argon compared to oxygen is likely due to the formation of oxygen vacancy complexes. This is consistent with the fact that annealing in an inert atmosphere promotes an increase in the concentration of such vacancies.

In addition to deep donor centers, shallow donor levels are also present in thin $(Y_{0.06}Ga_{0.94})_2O_3$ films, which may be caused by interstitial gallium atoms or more complex defect complexes involving gallium and oxygen vacancies. The activation energy of these

levels is approximately 0.25 eV and manifests differently depending on the heat treatment conditions.

The deeper donor levels in this region are likely associated with defect complexes containing oxygen vacancies. Since shallow donor levels have a shallower depth of occurrence compared to deep ones, they provide a higher concentration of free charge carriers, which explains the increased electrical conductivity of films annealed in a reducing hydrogen atmosphere.

Our studies have also shown that $(Y_{0.06}Ga_{0.94})_2O_3$ films that were not pre-annealed in an oxygen or argon atmosphere exhibit significantly higher conductivity after reduction annealing than films that underwent such pre-annealing at 1000 °C. This indicates that the formation of defects associated with increased electrical conductivity occurs much more readily in films with an incompletely formed structure. The observed effect is likely due to a lower energy barrier for the formation of intrinsic defects in an incompletely formed structure.

CONCLUSION

This study investigates the effect of preparation conditions and heat treatment on the morphological, structural, and electrical conductivity properties of thin films of $(Y_{0.06}Ga_{0.94})_2O_3$ obtained by radio-frequency ion plasma sputtering.

It was found that heat treatment significantly affects the microstructure of the films. In particular, annealing in an oxygen atmosphere promotes the formation of the most ordered polycrystalline structure, as confirmed by X-ray diffraction analysis. Morphological studies showed that grain growth and a decrease in surface roughness occur after annealing, indicating processes of recrystallization and structural ordering.

It has been shown that the electrical conductivity properties of the films significantly depend on the heat treatment conditions. The films after deposition are characterized by high resistivity and low activation energy for electrical conductivity. Annealing in oxygen and argon does not significantly affect electrical resistance at room temperature, but leads to a significant decrease at elevated temperatures. It was found that the thermal activation energy of electrical conductivity is approximately 0.87 eV for films annealed in oxygen and up to 1.38 eV for films annealed in argon, indicating different defect states of the material.

It has been established that annealing in a reducing hydrogen atmosphere leads to a significant decrease in resistivity and a reduction in activation energy to ~0.25 eV, which is due to an increase in the concentration of shallow donor centers.

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COMPLIANCE WITH ETHICAL STANDARDS

The authors declare that they have no competing interests.

AUTHOR CONTRIBUTIONS

Conceptualization, [O.B.]; methodology, [I.K., I.K.]; validation, [I.M.]; formal analysis, [I.K.]; investigation, [I.K., I.M., I.K.]; resources, [O.B.]; data curation, [O.B.]; writing – original draft preparation, [I.K.]; writing – review and editing, [I.M., O.B.]; visualization, [I.K., I.K.].

All authors have read and agreed to the published version of the manuscript.

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ВПЛИВ УМОВ ОДЕРЖАННЯ НА ЕЛЕКТРОПРОВІДНІ ВЛАСТИВОСТІ ТОНКИХ ПЛІВОК $(Y_{0.06}Ga_{0.94})_2O_3$

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АНОТАЦІЯ

Вступ. Оксид галію $\beta\text{-Ga}_2\text{O}_3$ є перспективним широкозонним напівпровідниковим матеріалом, який широко застосовується в оптоелектроніці та сенсорних системах. При цьому електропровідність тонких плівок значною мірою визначається їх структурною досконалістю, дефектною підсистемою та умовами післяростової обробки. У полікристалічних плівках міжзернові границі та дефектні комплекси суттєво впливають на механізми перенесення заряду. Метою роботи є дослідження впливу атмосфери відпалу на структурні, морфологічні та електропровідні властивості тонких плівок $(Y_{0.06}Ga_{0.94})_2O_3$.

Матеріали та методи. Тонкі плівки $(Y_{0.06}Ga_{0.94})_2O_3$ товщиною 0,3–1,0 мкм були отримані методом ВЧ іонно-плазмового розпилення на підкладках із плавненого кварцу. Після осадження проводився відпал у кисні та аргоні при температурах 1000–1100 °С, а також у водні при 600–650 °С. Структурні властивості досліджували методом рентгенівської дифракції, морфологію поверхні — за допомогою атомно-силової мікроскопії. Електропровідність вимірювали в температурному діапазоні 300–450 К, а енергію активації визначали з температурних залежностей провідності.

Результати. Рентгеноструктурний аналіз підтвердив формування плівок у моноклінній фазі $\beta\text{-Ga}_2\text{O}_3$ та підвищення ступеня кристалічності після відпалу в кисні. Встановлено, що свіжнанесені плівки мають високий питомий опір ($\rho > 10^{11}$ Ом·см), який зменшується при підвищенні температури та після відпалу. Для плівок, відпалених у кисні, енергія активації становить близько 0,87 еВ, тоді як для відпалу в аргоні вона досягає ~1,38 еВ (у діапазоні 300–400 К), що свідчить про формування глибших донорних рівнів, пов'язаних із кисневими вакансіями. Відпал у водні призводить до значного зниження питомого опору (~ 10^8 Ом·см) та енергії активації (~0,25 еВ), що зумовлено появою мілких донорних рівнів.

Висновок. Електропровідність тонких плівок $(Y_{0.06}Ga_{0.94})_2O_3$ визначається донорними рівнями дефектної природи, які формуються залежно від умов відпалу. Кисневе та аргонове середовище сприяють утворенню глибоких донорних центрів, тоді як відпал у водні призводить до формування мілких донорів і підвищення електропровідності.

Ключові слова: тонкі плівки, оксид галію, кристалічна структура, домішки, електропровідність.