Proceedings of the International Conference on Oxide Materials for Electronic Engineering (OMEE 2021)

Luminescence and Conductivity of β-Ga_2O_3 and β-Ga_2O_3:Mg Single Crystals

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The temperature-dependencies of the X-ray excited luminescence spectra were measured from unintentionally doped, annealed in oxygen and Mg-doped β-Ga_2O_3 crystals prepared by the floating zone growth method. The crystals exhibited ultraviolet, blue and green luminescence bands. It was established that the luminescence intensity is strongly dependent on resistivity of the crystal. For the as-grown sample with a high electron concentration, the main luminescence band at 300 K was blue. The blue luminescence band was observed over a wide range of temperatures, and the thermal quenching of luminescence was observed only at $T > 400$ K. The blue luminescence band was suppressed in high-resistance crystals, in particular in oxygen annealed and heavily doped β-Ga_2O_3 crystals with 1% Mg, while the UV band, on the contrary, increased and prevailed at low temperatures. Activation energies of separate luminescence bands and conductivity in the investigated crystals are calculated. Some correlations between conductivity and luminescence parameters were found and discussed to give hints for the further development of this material.

topics: gallium oxide, β-Ga_2O_3:Mg, luminescence, quenching

1. Introduction

Gallium oxide (β-Ga_2O_3) is a wide band-gap oxide semiconductor ($E_g = 4.9$ eV) that has unique conduction and tunable optical properties. β-Ga_2O_3 is a promising material for application in different areas including power electronic devices, solar-blind UV photodetectors, gas sensors, transparent electrodes and scintillation detectors [1–4].

Pioneering studies on the emission properties of gallium oxide were performed on β-Ga_2O_3 single crystals grown by the Czochralski's, Verneuil methods and floating zone techniques [5–7]. Typical emission spectra of gallium oxide show ultraviolet (UV), blue (BL) and green (G) luminescence bands [5–12]. Luminescence occurs as a result of radiative recombination of self-trapped excitons [6–12] or as a result of recombination of carriers by lattice defects [6–10]. That is why luminescence is a very useful tool for the detection and identification of impurities or intrinsic point defects in crystals. Despite the fact that much research have been devoted to the study of the gallium oxide luminescence, there is no unambiguous understanding of the luminescence nature at gallium oxide. Onuma [9] found a correlation between conductivity and luminescence in low-resistance β-Ga_2O_3:Si crystals and in high-resistance β-Ga_2O_3:N films. It is known that a magnesium impurity is used to compensate shallow donors and to obtain high-resistance gallium oxide [13, 14]. However, there are no studies in the literature devoted to the detailed study of luminescence and its correlation with conductivity in β-Ga_2O_3:Mg crystals. Therefore, further studies of luminescence are needed, including correlations between luminescence and other material properties that are determined by crystal defects.

The present work is devoted to the study of luminescence spectra, the dependence of the luminescence bands yield on temperature in unintentionally doped (UID) β-Ga_2O_3 crystals with high conductivity and high-resistance β-Ga_2O_3 crystals doped with magnesium, and the correlation between luminescence and electrophysical properties of these crystals.

2. Experimental details

Undoped and Mg-doped β-Ga_2O_3 crystals were grown by the floating zone technique in the air. Mg^{2+} impurity was added to the raw material for crystals growing in the form of MgO oxide. Samples for luminescence and conductivity studies in the form of plane-parallel plates were obtained by cliving a single-crystal of β-Ga_2O_3 along the (100) cleavage plane. Some samples of gallium oxide crystals were annealed in an oxygen atmosphere.

The β-Ga_2O_3 crystals with a thickness of 0.5 mm, loosely wrapped in Pt foil, were placed in a quartz ampoule, which was filled with oxygen after pumping air. Annealing was performed at an oxygen pressure of 1 atm and temperature of 1550 K for 50 h.

Luminescence studies were performed by irradiating β-Ga_2O_3 crystals with X-rays. A setup based on an SF-4A quartz monochromator was used to study the luminescence and its temperature dependences. X-ray excitation was provided by a microfocus X-ray tube URS-002 with a molybdenum anticathode (U = 45 kV, I = 0.3 mA) through the beryllium window of a cryostat. When studying the temperature dependences of X-ray luminescence, the appropriate spectral regions corresponding to the maxima of the emission bands were selected using a monochromator. Spectra were corrected for spectral sensitivity and monochromator dispersion.

Measurement of the electrical conductivity of low-resistance crystals was performed by the 4-probe method. The electrical contacts with the samples were made by RF-sputtering of titanium on opposite sides of the single-crystal plates. Measurement of the electrical conductivity of high-resistance crystals was performed by the 2-probe method using a voltmeter-electrometer B7-30.

3. Results

3.1. Luminescence spectra of β-Ga_2O_3 crystals

The studies of luminescence spectra were performed on as-grown (UID), annealed in an oxygen atmosphere and doped with Mg impurity β-Ga_2O_3 crystals. At 290 K, the luminescence spectrum of the UID β-Ga_2O_3 crystal (Fig. 1) was a broad complex luminescence band with a maximum of about 420 nm (≃ 2.95 eV). In the energy scale, the crystal luminescence spectrum was decomposed into elementary Gaussian curves. As follows from the decomposition (Fig. 1), the broad luminescence band is a superposition of three elementary bands with a half-width of about 0.45 eV. The most intense among them was the blue band with a maximum near 2.92 eV (≃ 425 nm). The UV band with a maximum at 3.25 eV (380 nm) has an intensity of ≈ 0.5 blue band intensity. The less intense (≃ 0.25) is the band in the green region of the spectrum at 2.5 eV (495 nm).

At 90 K (Fig. 1b), a small increase in the integral intensity and redistribution of the intensity between the elementary bands are observed. The UV band becomes the dominant band in the luminescence spectrum, while the intensity of the blue band changes slightly. At the same time, the intensity of the green luminescence band of 2.5 eV decreases.

After annealing of the UID β-Ga_2O_3 crystals in an oxygen atmosphere, the luminescence yield at 290 K decreased by about half, and the maximum luminescence was shifted to 400 nm. At the same time, in the red region of the spectrum (1.5–1.8 eV), the intensity of red luminescence of uncontrolled

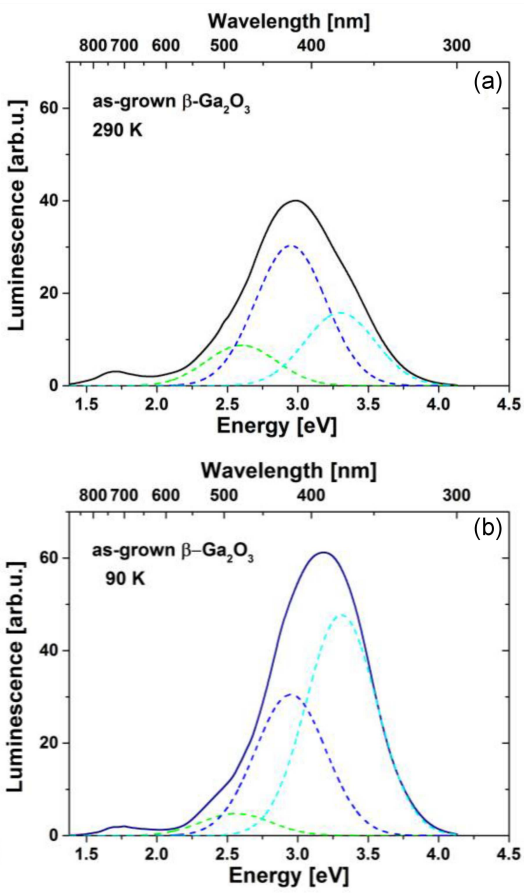

Fig. 1. Luminescence spectra of as-grown (UID) β-Ga_2O_3 crystals at 290 K (a) and 90 K (b).

chromium impurity (∼ 700 nm) increased significantly [15, 16]. At 90 K (Fig. 2b), the integral luminescence yield increases by almost an order of magnitude, and the maximum luminescence shifts towards the UV region. As can be seen in the appropriate inset in Fig. 2, the shift is the result of a strong increase in the intensity of the UV luminescence band.

The luminescence yield of β-Ga_2O_3:0.05%Mg (Fig. 3a) at 290 K is the same as in as-grown UID β-Ga_2O_3 crystals, but the maximum of the luminescence is shifted to a long-wavelength up to 450 nm. The green band (≃ 2.6 eV) is the main in the luminescence spectrum, while the intensity of the UV band does not exceed 2% of the maximum intensity. The inset shows that after doping with Mg, the luminescence in the red and green spectrum regions increases, and the UV luminescence band decreases. At 90 K (Fig. 3b), the integral luminescence yield increases, and the luminescence maximum shifts to the UV region. The shift is a consequence of an increase in the intensity of the blue and UV luminescence bands. In contrast to the as-grown crystal, in the luminescence spectrum of the β-Ga_2O_3:0.05%Mg crystal at 90 K, the main band is blue.

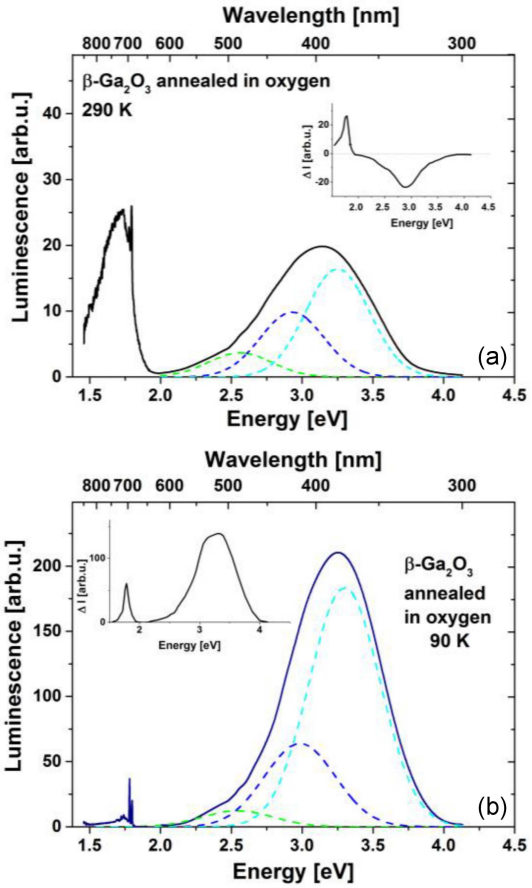

Fig. 2. Luminescence spectra of annealed in the oxygen β-Ga_2O_3crystals at 290 K (a) and 90 K (b). The insets present the difference spectra of annealed and as-grown β-Ga_2O_3 crystals.

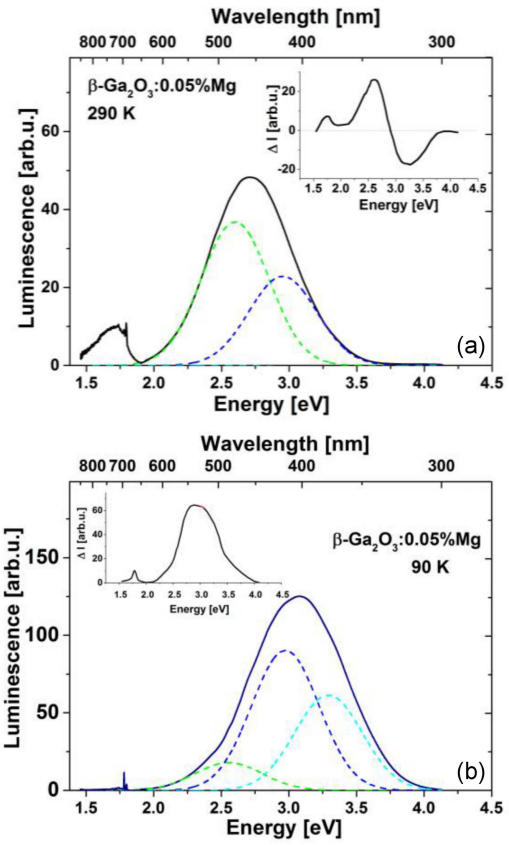

Fig. 3. Luminescence spectra of as-grown β-Ga_2O_3:0.05%Mg crystals at 290 K (a) and 90 K (b). The insets present the difference luminescence spectra of β-Ga_2O_3:0.05%Mg and β-Ga_2O_3 crystals.

In the β-Ga_2O_3:1%Mg crystal, the yield of intrinsic luminescence is low (in Fig. 4a it is increased by 10 times), and the main luminescence band at room temperature is the luminescence of uncontrolled chromium impurity. At 90 K (Fig. 3b), the intrinsic luminescence output increases by almost an order of magnitude, and the luminescence maximum shifts towards the UV region. The inset shows that with the increase in the concentration of magnesium impurity, an increase in the intensity of chromium luminescence and UV luminescence band are observed, as well as a decrease in the intensity of blue luminescence.

3.2. Temperature dependences of the luminescence yield

Figure 5a–d shows the temperature dependences of the intensity of the UV, blue and green luminescence bands for various crystals. The UV luminescence in the as-grown β-Ga_2O_3 crystal (squares) was observed over a wide temperature range. As temperature rises from 90 to 150 K, there is a slight increase followed by a decrease in intensity. At temperatures above 300 K, complete thermal quenching of the luminescence occurs. The change in the luminescence intensity is well described by Mott's law. The best agreement of the experimental results with the dependence $I = I_0/(1 + B\exp(-E_q/(k_BT)))$ was obtained for $E_q \simeq 0.16$ eV. The yield of the blue 425 nm luminescence band (blue squares) behaves in a similar way. As temperature rises from 90 to 150 K, a slight increase in the luminescence yield was observed first, then a plateau, and at higher temperatures $T > 370$ K there was a sharp decrease and complete temperature quenching of luminescence at 540 K. Fitting at low temperatures (150–300 K) gives energy E_q close to 0.1 eV. In the temperature quenching region (370–500 K) the best agreement was obtained at $E_q = 0.48$ eV. The temperature dependence of the green luminescence band differs from the temperature dependence of the blue and UV bands. The green band has a low luminescence yield at 90 K and increases when heated to 370 K. At higher temperatures, a decrease in the intensity and complete quenching of the green luminescence is observed. Calculation of the activation energy of the thermal quenching process in the temperature range $T > 360$ K gives the values of $E_q = 0.63$ eV.

TABLE I

Conductivity, activation energy of conductivity and energy of temperature quenching of luminescence for in different β-Ga_2O_3 crystals.

	Conductivity [Ω^{-1}cm^{-1}]	Activation energy of conductivity E_a [eV]	Energy of luminescence temperature quenching E_q [eV]		
			UV luminescence	Blue luminescence	Green luminescence
as-grown β-Ga_2O_3	2.5	0.02–0.03	0.16	0.48	0.63
annealed in O_2 β-Ga_2O_3 (UID)	10^{-8}–10^{-10}	0.8	0.08	0.1	0.08
as-grown β-Ga_2O_3:0.05%Mg	10^{-5}–10^{-6}	0.1–0.2	0.1	0.4	0.45
as-grown β-Ga_2O_3:1%Mg	10^{-12}–10^{-14}	0.84	0.06	0.08	0.08

For the as-grown β-Ga_2O_3:0.05%Mg crystal (Fig. 5c), similar temperature dependences were obtained for the blue and green luminescence bands. However, as in the case of the UID crystal, the quenching of the UV luminescence band starts at lower temperatures. Temperature quenching energies are presented in Fig. 5c and Table I.

Quite different temperature dependences of the luminescence bands were obtained for high-resistance gallium oxide crystals annealed in an oxygen atmosphere or doped with magnesium β-Ga_2O_3:1%Mg. In β-Ga_2O_3 crystals annealed in an oxygen atmosphere (Fig. 5b), the maximum intensity of all luminescence bands is observed at 90 K. The increase in temperature is accompanied by a gradual decrease in the luminescence intensity. The change in intensity with temperature is also well described by Mott's law. The quenching activation energy E_q is about 0.08–0.1 eV for all luminescence bands. In the β-Ga_2O_3:1%Mg crystal, the temperature dependences of the UV, blue, and green luminescence bands yield are similar to those observed in an annealed crystal. The obtained activation energies for the thermal quenching of luminescence are slightly lower than for the crystal annealed in oxygen and are in the range of 0.06–0.08 eV.

3.3. Electrical properties

The electrical conductivity of as-grown UID β-Ga_2O_3 single crystals was high and reached 2.5 Ω^{-1} cm^{-1} at 290 K. The activation energy of conductivity E_a, calculated in the linear region from the dependence $\ln(\sigma) = f(1000/T)$, was about 0.02–0.03 eV. The concentrations of uncompensated donors in the as-grown crystals were in the range of $(N_d - N_a) > 1 \times 10^{18}$–$1 \times 10^{19}$ cm^{-3}.

Annealing of low-resistance as-grown β-Ga_2O_3 crystals with a thickness of $\sim$ 0.5 mm in an oxygen atmosphere for 50 h at a temperature of 1550 K led to a decrease in the conductivity to 10^{-8}–10^{-10} Ω^{-1}cm^{-1}. The thermal activation energy, calculated from the temperature dependence of conductivity, in the high-resistance samples was $E_a \simeq 0.8$ eV.

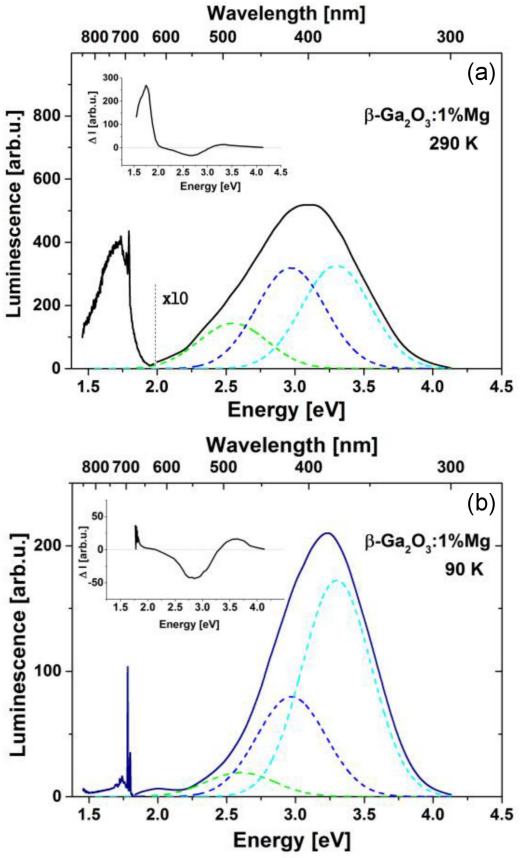

Fig. 4. Luminescence spectra of the as-grown β-Ga_2O_3:1%Mg at 290 K (a) and 90 K (b). The insets present the difference luminescence spectrum of β-Ga_2O_3:1%Mg and β-Ga_2O_3:0.05%Mg crystals.

In doped as-grown β-Ga_2O_3:0.05%Mg crystals with a low concentration of magnesium, a sufficiently high conductivity was obtained. At 300 K, the conductivity of the grown β-Ga_2O_3:0.05%Mg crystals was 10^{-5}–10^{-6} Ω^{-1}cm^{-1}, which is much less than the as-grown crystals, but much higher than the conductivity of crystals annealed in an oxygen atmosphere (Table I). The activation energy of the conductivity of the grown β-Ga_2O_3:0.05%Mg crystal was E_a = 0.1–0.2 eV. The electrical

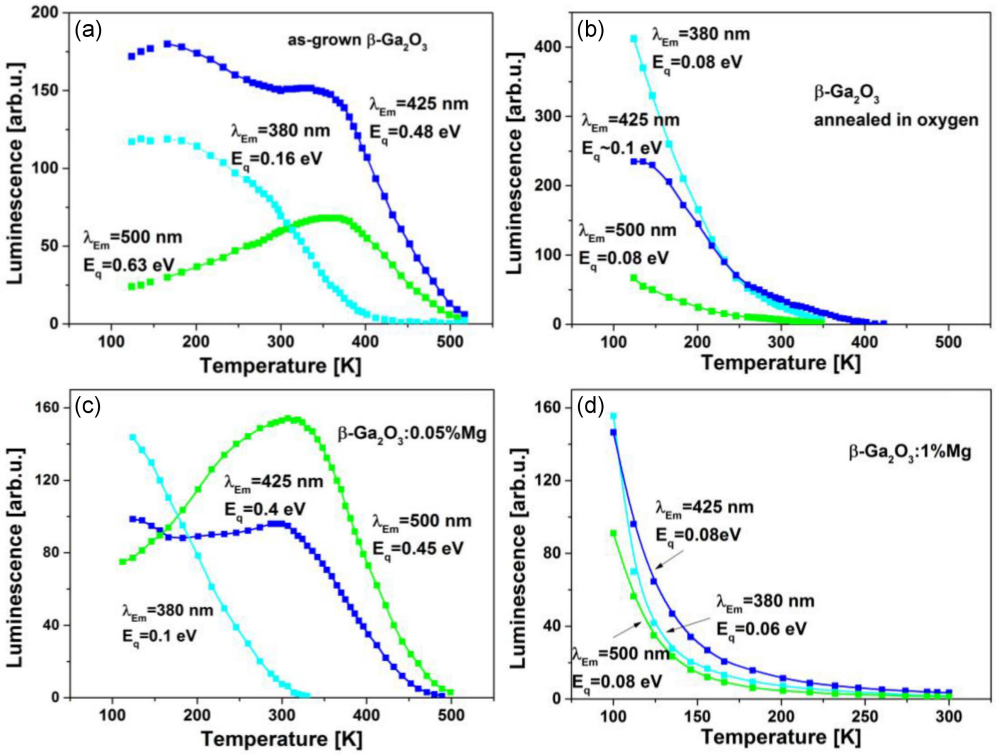

Fig. 5. Temperature dependences of UV, blue and green luminescence bands: as-grown β-Ga_2O_3 (a), β-Ga_2O_3 annealed in oxygen (b), β-Ga_2O_3:0.05%Mg (c) and β-Ga_2O_3:1%Mg (d) crystals.

conductivity value is between the value for UID β-Ga_2O_3 and β-Ga_2O_3:1%Mg. It is known that doping of β-Ga_2O_3 with Mg impurity leads to the creation of acceptor levels [13, 17]. Apparently, in the β-Ga_2O_3:0.05%Mg crystals the concentration of Mg acceptors is close to that of shallow donors. They compensate the part of the shallow donors. Thus, the conductivity of these crystals is controlled by deeper ($E_{\simeq}$0.1–0.2 eV) Ga_i donors.

In contrast to as-grown β-Ga_2O_3:0.05%Mg crystals, as-grown β-Ga_2O_3:1%Mg crystals were high-resistivity, their conductivity was in the range ($\sim 10^{-12}$–10^{-14} Ω^{-1}cm^{-1}). The conductivity activation energy calculated from the temperature dependence of the resistance for these crystals was $E_a = 0.84$ eV.

4. Discussion

Correlations between conductivity and luminescence were previously observed on Si-doped β-Ga_2O_3 crystals and N-doped β-Ga_2O_3 films [9]. From the above results it follows that correlations between luminescence spectra and electrical properties are also observed in UID, oxygen annealed and Mg-doped β-Ga_2O_3 crystals.

The main luminescence band in the as-grown UID β-Ga_2O_3 crystals with high conductivity was the blue luminescence band with a maximum of about 425 nm. The blue band of luminescence did not change much with temperature, and thermal quenching of luminescence was observed only at $T > 400$ K. The as-grown β-Ga_2O_3:0.05%Mg crystals also had high conductivity. In such β-Ga_2O_3:0.05%Mg crystals, the blue luminescence band also prevailed in a wide temperature range from 90 to 400 K.

The blue luminescence band of β-Ga_2O_3 is most often attributed to recombination of donor–acceptor pairs (DAP) or electrons from the conduction band with holes localized on the defects [6–9]. Donors are formed by silicon [14] or interstitial gallium [18, 19]. Before excitation, donors are filled with electrons and acceptor levels are empty. When the β-Ga_2O_3 crystal is irradiated with X-rays, free electrons and holes are formed. The holes are immediately trapped by negatively charged defects (V_{Ga}, $V_{Ga}V_O$, Mg_{Ga}) [17] or self-trapped. As a result, DAP filled with electrons and holes are formed. Recombination of electrons with holes in DAP gives luminescence in the blue region. Due to the fact that there are different donors and acceptors, the energy of DAP radiation can be in a wide range of energies from 2.6 to 3.3 eV. Since the donors in conductive β-Ga_2O_3 crystals are filled with electrons, the blue luminescence band quenching is due to thermal delocalization of holes from the acceptor levels. The value of the temperature quenching activation energy $E_q \simeq 0.5$ eV calculated from the curve of the blue luminescence yield temperature dependence correlates with the position of the energy levels created by gallium vacancies or magnesium impurities [13, 20, 21].

The concentration of donors in high-resistive β-Ga_2O_3 crystals is low and most of them are ionized even at low temperatures. Therefore, the number of DAP involved in radiative recombination is small and the yield of the blue luminescence band in such crystals will also be low. When such a β-Ga_2O_3 crystal is irradiated with X-rays, electrons are trapped by ionized donors, and holes are trapped by negatively charged acceptor defects. The temperature quenching of the blue luminescence in such high-resistive crystals is primarily due to thermal delocalization of trapped electrons from the shallow donors. Therefore, the quenching energy is much lower than that in low-resistance crystals. Studies of thermally stimulated luminescence and conductivity [18, 21] have shown that annealing of crystals in an oxygen atmosphere or doping with magnesium leads to a decrease in the concentration of shallow (0.1–0.25 eV) trap levels created by the ionized donors in gallium oxide.

The main luminescence band in high-resistive crystals is the UV band at 380 nm. The UV luminescence band is well described by the model of radiative recombination of self-trapped excitons. UV luminescence has a significant Stokes shift, high quantum yield at low temperatures, a wide elementary band with a half-width of $\simeq 0.45$ eV, decay time less than 10^{-6} s [4]. It does not depend on impurities (its intensity can only decrease with increasing crystal defects or doping, as we can see). Self-trapping occurs as a result of the deformation interaction of electronic excitations with the acoustic oscillations of the lattice [11, 12]. The thermal quenching energy $E_q \simeq 0.08$ eV of UV luminescence is close to the phonon energy of galluim oxide and may result from thermal destruction of autolocalized states [12]. The low quantum yield of the UV luminescence band in crystals with high conductivity may be the result of Debye–Hückel screening [9], or due to the high competition of the carrier recombination channel through DAP. It is also possible that the low-intensity luminescence observed in such crystals in the UV region of the spectrum is due to the recombination in DAP formed by shallow donors and acceptors [6–9].

A wide band of luminescence in the green part of the spectrum was mainly observed in high-resistive samples, when there is a strong compensation of donors. The green luminescence band is the main band in Mg-dopped crystals. Doping β-Ga_2O_3 with an impurity of divalent magnesium also leads to an increase in the luminescence output in the green region of the spectrum. One of the possible models of the green luminescence center is the DAP model, in which one of the components forms deep levels of oxygen vacancy V_O and the hole component is $V_{Ga}V_O$, or Mg_{Ga}. However, additional research is needed to understand the nature of the green luminescence band in gallium oxide.

5. Conclusions

Study of luminescence spectra under X-ray excitation and temperature dependencies of the luminescence yield of individual luminescence bands in undoped, annealed in oxygen and Mg-doped β-Ga_2O_3 crystals are presented. It has been shown that blue luminescence predominates in highly conductive gallium oxide crystals. On the contrary, in high-resistance crystals obtained as a result of annealing in an oxygen atmosphere or as a result of doping with a magnesium impurity, the UV luminescence band predominates. The blue luminescence band is due to recombination of carriers in the DAP formed by shallow donors and acceptor levels. Shallow donors are formed by uncontrolled impurities of Si, Sn, Ge and intrinsic defects such as interstitial gallium. Acceptors in β-Ga_2O_3 are formed by gallium vacancies V_{Ga}, bivacancies $V_{Ga}V_O$ and divalent metal atoms Me^{2+}, in particular by magnesium impurities. The high temperature quenching of the blue luminescence band is due to the delocalization of holes from the acceptor levels. The quenching of the blue band of luminescence in high-resistive crystals is due to the thermal delocalization of electrons from the donors' levels. The UV luminescence band, which dominates in high-resistance samples at low temperatures, is due to the radiative recombination of self-trapped excitons. The temperature quenching of the UV luminescence band is due to the destruction of self-trapped excitons. Some correlations are found between conductivity and luminescence parameters. UID β-Ga_2O_3 crystals have the highest conductivity. In high-resistance crystals obtained by doping 0.05%Mg^{2+} and annealing in an oxygen atmosphere, the conductivity decreased by at least 10^5 and 10^8 times, respectively. The most high-resistance crystals were obtained by doping with 1%Mg^{2+} impurity.

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