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Controlling of the charge states: Irradiation and Annealing in Oxidizing and Reducing Atmospheres as a Method of Crystal Change and Characterization

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Abstract. The results of the effect of annealing and irradiation treatments on the optical properties of $Y_3Al_5O_{12}$, $YAlO_3$, $SrLaGa_3O_7$, $LiNbO_3$, $Gd_3Ga_5O_{12}$, $LaGaO_3$, ZnSe and LiF single crystals are described. Recharging processes of uncontrolled impurities (e.g. Fe^{3+}, Fe^{2+} and Mn^{2+}), recharging processes of active ions (e.g. Nd^{3+}, Dy^{3+}, Cr^{4+}, Cr^{3+} and Ce^{3+}), and types of color centers produced in the crystals after a particular irradiation or annealing treatment and changes in luminescence are presented.

Keywords: absorption, additional absorption, electron spin resonance, gamma and electron irradiation

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1. INTRODUCTION

It is known that color centers (CC) in oxide compounds, like Nd: $Y_3Al_5O_{12}$ (Nd: YAG), Er: YAG, Nd: $Gd_3Ga_5O_{12}$ (Nd: GGG), Nd: $SrLaGa_3O_7$ (Nd: SLGO), Cr: SLGO, Nd: $YAlO_3$ (Nd: YAP), Pr: YAP and Er: YAP, produced by the ultraviolet part of pumping lamp's light spectrum as well as by the bombardment of various types of ionizing particles (gamma-rays, electrons, protons etc.), adversely influence the output characteristics of the solid-state lasers [1-3]. Common methods of characterization of laser crystals may not give a precise answer to the question: which is the performance of lasers in conditions of strong external radiation and thermal fields. This problem is very important for laser devices working, for example, in outer space.

The aim of this paper is to describe the possibilities of the method for characterization of the compounds and, may be, for changing their optical properties.

2. SOME REMARKS ON THE INTERACTION OF THE IONIZING RADIATION WITH CRYSTALS

Various kinds of the ionizing radiation interact with crystals in different ways.

2.1 Gamma rays

2.1.1 Recombination of the ions

Gamma photons interact with atoms of the crystal lattice by means of the photo-effect and Compton effect and, for energies higher than 1.021 MeV, by the annihilation process. They become the source of secondary electrons which can recombine with already existing CC (after growth process) or active impurities, causing their recharging. For gamma rays from ^{60}Co source, the prevailing effect in interaction with atoms of e.g. YAG crystal is the Compton effect, so the maximum electron energy is about 900 keV. Because the energy spectrum is practically flat, then average energy of the Compton electron is about 450 keV, so one can expect about 20-30 secondary electrons to be emitted in one interaction of a photon with the crystal.

2.1.2 Additional ionization

Gamma rays incident on the crystal or some of secondary electrons can also ionize CC or impurities leading to the recharging process of opposite type to the previous case.

2.2 Electrons

Effect of the electron irradiation is essentially comparable with the effect of gamma irradiation of the same energy. The only difference is that electrons from the accelerator are initially mono-energetic while those from Compton scattering of gamma rays have continuous spectrum. In the case of electron irradiation, the average energy registered locally in the volume element of the sample varies with depth due to degradation of energy of the initial electron, while for gamma rays, the local energy of the secondary Compton electrons essentially does not depend on depth. The cross-section for recombination of ions depends on the electron energy spectrum. Therefore, some difference in the recombination rate should be observed in the crystal sample for different depths of the penetrating electrons.

Electrons can interact with atoms of a crystal lattice via elastic scattering too. This type of interaction leads to formation of the Frenkel defects.

2.3 Protons

Fast protons penetrating the crystal sample lose continuously their energy. Part of this energy is transferred to the secondary delta electrons which become the source of the recharging effect of ions present in the crystal. The energy spectrum of these secondary electrons differs from those for electron and gamma incident particles. Therefore the effective probability of the recombination should be different than for primary or secondary electrons.

The interaction of the proton with ions inside the crystal lattice can also lead to the ionization effect which causes recharging of ions towards the higher valency.

Fast proton can also effectively interact with atoms via elastic scattering process giving rise in the formation of Frenkel defects. Because in the interaction of protons the momentum transfer to the recoil nucleus is much larger than in the case of fast electrons, the former process should be much more effective than the latter.

3. EXPERIMENTAL

The following crystals were investigated: $Y_3Al_5O_{12}$ (YAG), $YAlO_3$ (YAP), $SrLaGa_3O_7$ (SLGO), $LiNbO_3$ (LN), $Gd_3Ga_5O_{12}$ (GGG), $LaGaO_3$ and ZnSe and LiF. They were obtained in the Institute of Electronic Materials Technology, Institute of Physics Polish Academy of Sciences and Institute of Physics Military University of Technology in Warsaw.

3.1 Irradiation of the crystals

Using various ^{60}Co sources the crystals were irradiated by γ-rays in the Institute of Chemistry and Nuclear Technology (ICNT) in Warsaw and Institute of Atomic Energy in Świerk, Poland. For the electron irradiation 300 keV or 1 MeV beams from the Van de Graaf accelerator of the ICNT were used, while for irradiation of 21 MeV protons a beam from compact isochronous proton cyclotron installed in the Soltan Institute of Nuclear Studies, Świerk, was applied.

The dose of γ-irradiation was varied from 10^2 to 10^7 Gy, while fluency of electrons varied from 10^{14} to $5*10^{16}$ particles/cm^2 and protons from $5*10^{12}$ to 10^{16} particles/cm^2.

3.2 Annealing procedures

Annealing was performed in three regimes: (i) - thermal relaxation by annealing in the air at 400^{o}C for YAG and at 800^{o}C for LN for 3 hours in order to remove radiation defects, (ii) - annealing in the oxidizing atmosphere by heating-up at 1400^{o}C (or 1100^{o}C) for 3 hours in the air in order to change the defect structure of a crystal and (iii) - annealing in a reducing atmosphere by heating in a mixture of hydrogen and nitrogen at 1200^{o}C for 0.5 h for the same purpose.

3.3 Spectroscopic investigations

Optical transmission spectra were recorded before and after each irradiation or thermal treatment of the samples by LAMBDA-2 PERKIN-ELMER, ACTA VII BECKMAN and FTIR 1725 PERKIN-ELMER spectrophotometers. The induced additional absorption (AA) was calculated from the formula:

$$\Delta K = 1/d \ln(T_1/T_2) \tag{1}$$

where K is the absorption, d is the sample thickness, and T_1 and T_2 are the optical transmissions of a sample before and after irradiation or annealing treatment, respectively.

4. RESULTS AND DISCUSSION

4.1. Absorption

Fig. 1 shows additional absorption of Cr doped SLGO (Fig. 1a) and Pr, Er and Nd doped YAP (Fig. 1b, c) crystals after four different kinds of treatments: γ-irradiation of as-grown crystals (Fig. 1a, curve 2; Fig. 1b, curve 1; Figure 1c, curves 1 and 2), annealing of as-grown crystals in the air (Fig. 1a, curve 1; Fig. 1b, curve 3), annealing in reducing atmosphere (Fig. 1b, curve 2) and proton irradiation (Fig. 1a, curve 3; Fig. 1c, curve 3).

For both types of crystals (YAP and SLGO) a large additional absorption (large ΔK values) was observed after different treatments. As one can see from Fig. 1a, the influence of temperature (annealing in the air) and ionizing rays (gamma and protons) on the absorption spectrum of Cr: SLGO crystal is of the same type in the range of Cr transitions (500-800 nm). Negative value of AA in this range suggests that ionization of active Cr^{3+} ions takes place during both types of treatments, resulting in an increase in concentration of Cr^{4+} ions. However, these two types of treatments differ from each other in producing radiation defects.

Irradiation with gamma and proton rays leads to an increase in CC intensity with a maximum at about 380 nm but gamma gives larger AA values. This CC is associated with oxide vacancies in Cr: SLGO single crystal, as it was found earlier [4]. Moreover, for both types of the above treatments the AA band appears at about 270 nm. This CC is attributed to $Ga^{3+} \rightarrow Ga^{2+}$ reaction leading to the shift of a short-wave absorption edge [4].

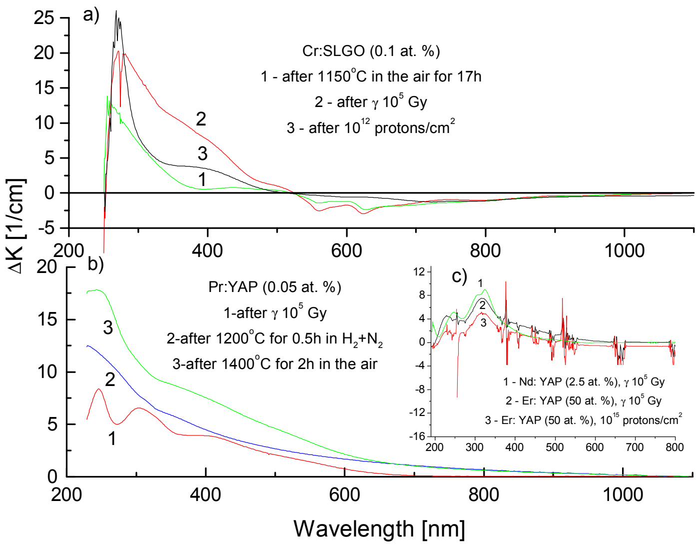

Fig. 1. AA bands in Cr: SLGO (a), Pr: YAP (b) and, Nd: YAP and Er: YAP (c) for different types of treatments

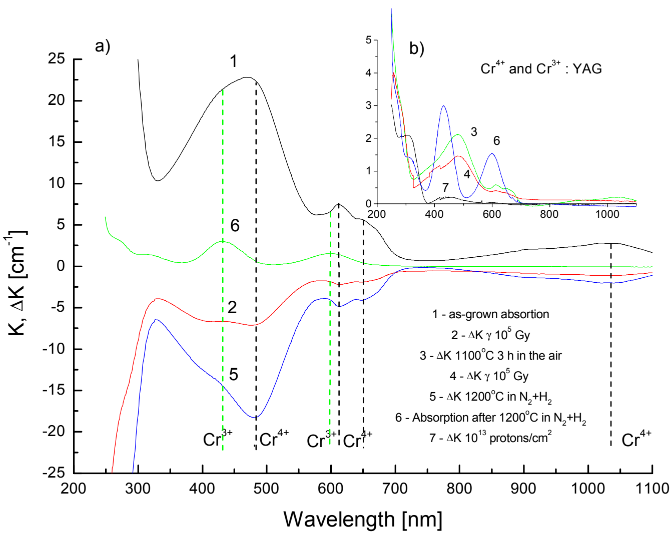

Fig. 2. Absorption (curves 1 and 6) and additional absorption (curves 2-5 and 7) of Cr^{4+} doped YAG crystal after subsequent treatments: gamma irradiation with a dose of 10^5 Gy (2), annealing at 1100°C in air for 3h (3), gamma irradiation with the same dose as previously (4), annealing at 1200°C in N_2+H_2 mixture for 0.5h (5) and, proton irradiation with a fluency of 10^{12} protons/cm^2 (7)

Fig. 1b shows that annealing of Pr: YAP crystal in different atmospheres leads to similar AA bands as compared to γ-rays. Usually additional absorptions obtained after annealing and irradiation (especially for annealing performed after irradiation) have opposite values. Annealing in the air leads to an increase in the valency of uncontrolled or active impurity, while gamma irradiation and annealing in reducing atmosphere lead to a decrease in the valency of the impurity due to recombination of ions with Compton secondary electrons (or reduction) [2].

Fig. 1c shows changes in the absorption of Er: YAP and Nd: YAP after gamma irradiation with the same dose of 10^5 Gy (curves 1 and 2) and changes after proton irradiation with a fluency of 10^{14} protons/cm^2 for Er: YAP (curve 3). Clearly, all the changes are of the same type, i.e. the appearance of CC is of the same type.

Fig. 2 shows the absorption (K) and changes in the absorption (ΔK) (compare curves 1 and 2) for Cr^{4+} (Cr^{4+}: YAG crystal, curve 1) and Cr^{3+} doped YAG crystals (after annealing of Cr^{4+}: YAG crystal in reducing atmosphere). The as-grown Cr^{4+}: YAG crystal was black, while the Cr^{3+}: YAG was green. As it is seen from absorption curves both crystals contain the second type of dopant (Cr^{4+}: YAG crystal contains also Cr^{3+} and Cr^{3+} :YAG crystal contains also Cr^{4+}). The dashed lines mark the positions of absorption bands characteristic for a given type of the active dopant.

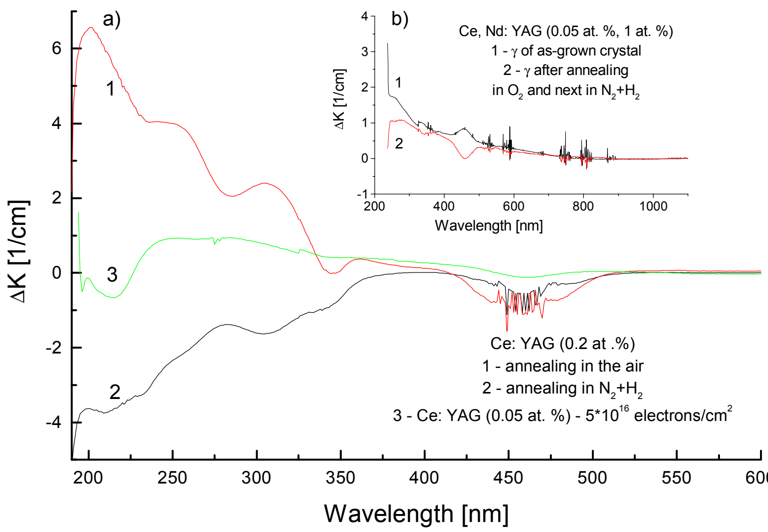

Fig. 3. AA bands in Ce: YAG (0.2 at. % and 0.05 at. %) (a) single crystals after different types of annealing procedures (curves 1, 2) and electron irradiation with a fluency of $5*10^{16}$ cm^{-2} (curve 3) and in Ce, Nd: YAG (0.05 at. %, 1 at. %) (b) after γ-irradiation with a dose of 10^5 Gy

Thus, γ-irradiation of as-grown crystal (curve 2) and annealing of the crystal in reducing atmosphere (curve 5) lead to a decrease in concentration of Cr^{4+} ions due to ion recombination effect ($Cr^{4+} \rightarrow Cr^{3+}$) and reduction, respectively. Annealing in the air (curve 3) and subsequent γ-irradiation (curve 4) lead to an increase in Cr^{4+} ions concentration due to the ionization effect ($Cr^{3+} \rightarrow Cr^{4+}$). However the explanation of proton interactions with crystals is more complicated. After annealing the crystal in the reducing atmosphere (when majority of Cr ions are trivalent), protons interact similarly as gamma quanta, producing more Cr^{3+} ions due to δ–electrons emitted along the proton trajectory. As it was mentioned above, certain amount of Cr^{4+} ions exists in the Cr^{3+}: YAG crystals. These Cr^{4+} ions recombine with δ–electrons in the same way as secondary electrons created by gamma rays in Compton process.

In the case of chromium doped YAG crystals mainly recharging processes of chromium ions were observed. Akhmadulin et al. [5] showed that depending on the applied annealing process of YAG crystals (annealing in oxidizing or reducing atmosphere) γ–irradiation may produce two different types of defects: V_o – type vacancies and O^- - type holes (the band with a peak at 385 nm) for the crystal annealed in an oxidizing atmosphere, while for a crystal annealed in a reducing atmosphere, F^- centers.

Fig. 3a shows changes in absorption spectra (ΔK) after two types of annealing treatments for Ce:$Y_3Al_5O_{12}$ (Ce: YAG) single crystal. Clearly, the influence of both types of annealing is opposite to each other (curves 1 and 2). Moreover the recharging effect is seen for Fe^{3+} (a band centered at 253 nm) and Ce^{3+} (bands centered at 338 and 458 nm, Fig. 3a and Fig. 3b) ions. Radiation can influence the absorption spectrum oppositely, depending on the previous annealing treatment for irradiated crystal as it is shown in Fig. 3b (curves 1 and 2). Electrons interact with active (Ce) and uncontrolled (Fe) ions recharging them as it is seen in Fig. 3a (curve 3). The Ce: YAG crystal doped with a high concentration of Ce (0.2 at. %) is strongly defected.

Fig. 4 presents changes in absorption spectrum of (a) Nd: GGG and (b) pure YAG single crystals under irradiation with γ-rays as compared to annealing in reducing atmosphere (after annealing of Nd: GGG crystal in reducing atmosphere secondary polishing was necessary). As one can see from the figure, if no oxide vacancies are present in a crystal, then the AA band (which appears in the absorption spectrum of the crystal after treatment with γ-rays and annealing in reducing atmosphere) has the same shape (see curves 1 and 2, Fig. 4b and, for comparison, curves 1 and 2, Fig. 4a). The presence of oxide vacancies in YAG crystal is observed as CC with weak maximum at about 450 nm (see curve 3, Fig. 4b), while in GGG crystal as CC with maximum at about 340 nm.

Fig. 5 shows changes in the absorption spectrum of $LiNbO_3$ single crystal doped with (a) 1 at. % Dy and (b) 0.03 at. % Er after γ-irradiation with a dose of 10^5 Gy. $LiNbO_3$ single crystals are in general poorly affected by gamma or proton irradiations than YAP, SLGO, YAG or GGG crystals. The additional absorption is only 0.4 cm^{-1} in contrast to other crystals where it may be equal to even 25 cm^{-1} (see, for example, Fig. 1a).

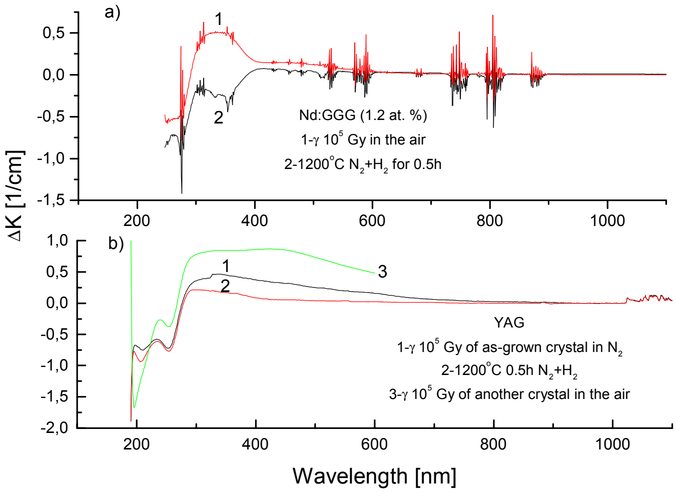

Fig. 4. AA bands in Nd: GGG (a) and YAG (b) single crystals after γ-irradiation (curve 1 in Fig. 4a and curve 1 and 3 in Fig. 4b) as compared to annealing in reducing atmosphere (curve 2 in Fig. 4a and curve 2 in Fig. 4b)

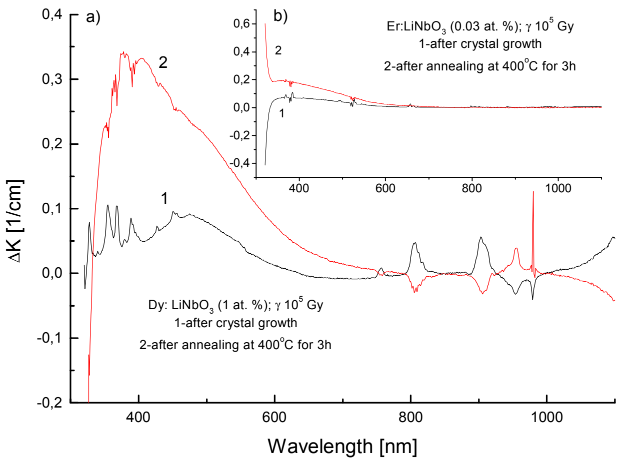

Fig. 5. AA bands in Dy doped (a) and Er doped (b) single crystals after γ-irradiation performed before and after annealing in the air at 400°C

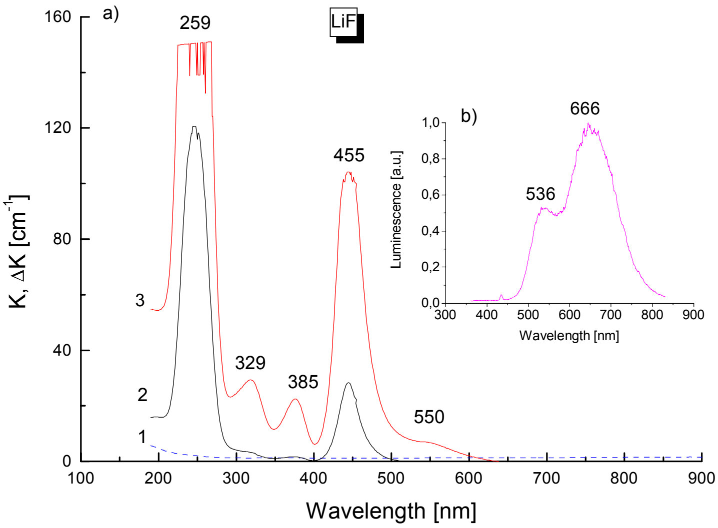

Fig. 6. Absorption (1), additional absorption after γ-irradiation with a dose of 10^5 Gy (2) and 10^6 Gy (3) – (a) and excited with 442 nm photoluminescence (b) of LiF single crystal

As one can see from Fig. 5 for a crystal which is thermally unstable, γ-irradiation of the previously annealed crystal gives larger values for AA intensity than for as-grown crystal. One of the possible reason for this behavior may be that annealing of LN crystals was performed at relatively low temperature.

Arizmendi et al. [6] have shown that defects created in $LiNbO_3$ crystal after annealing and irradiation differ from each other. In both types of treatments polarons with energy of 1.6 eV, trapped holes in the case of irradiated crystals and F and F^+ centers in the case of reduced crystals were observed.

In Fig. 5a recharging process of dysprosium ions is also seen for Dy^{3+}: LN crystal (see changes in absorption spectrum for wavelengths greater than 800 nm). This suggests that Dy^{2+} ions are probably present in the as-grown crystals [7].

3.2. Luminescence

Fig. 6 shows absorption (curve 1), additional absorption after γ-irradiation with a dose of 10^5 Gy (curve 2), 10^6 Gy (curve 3) and luminescence after this irradiation (curve 4) observed for LiF single crystal. Obviously, after γ-irradiation of as grown LiF crystal CC peaks appear at 259, 329, 385, 455 and 550 nm. The band at 455 nm is attributed to [F_2] center, [--|2e|], while the band at 550 nm is attributed to [F^{2+}] center, [--|e|] [8]. The intensity of these CC's is much greater than that for other crystals (e.g. for 259 nm band it is as high as 160 cm^{-1}). It depends also on the dose value (compare curves 2 and 3), moreover, for γ-irradiated crystals strong emission at 536 and 664 nm is found after exciting the crystal with λ_{ex}=442 nm (laser excitation). Exciting with 455 nm gives two bands equal in intensity while exciting with 385 nm gives much higher intensity of 536 nm band in comparison with 664 one. CC's in LiF crystal are often applied as Q-switches in laser systems [9]. Such behavior of the crystals suggests that ionizing radiation can radically change optical properties of a crystal.

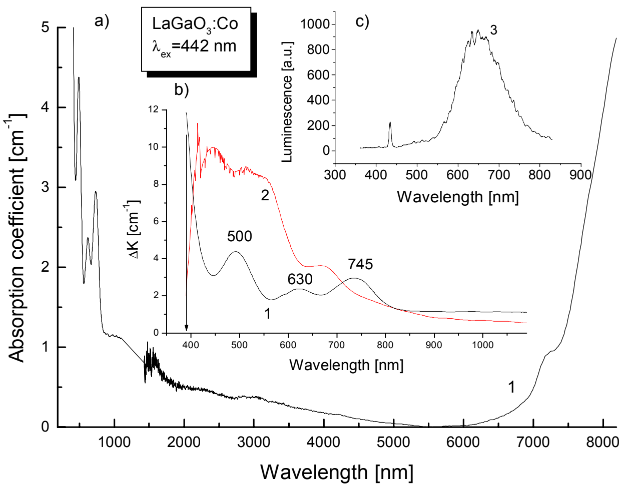

Fig. 7. Absorption of as-grown crystal (1), additional absorption (2) and photoluminescence of γ-irradiated Co: $LaGaO_3$ single crystal (dose $4*10^5$ Gy)

Fig. 7 presents absorption (curve 1), additional absorption (curve 2) and photoluminescence (curve 3) of Co:$LaGaO_3$ single crystal. The absorption spectrum shows transitions in Co^{2+} and Co^{3+} mixed system seen in the range of VIS and near IR. Co^{3+} has got two excited states in visible part of the spectrum, $^1T_1$ (about 667-769 nm) and $^1T_2$ (about 455-500 nm) and one in IR (about 1600 nm). The as-grown Co^{3+}: $LaGaO_3$ crystal does not show any luminescence in visible range. After γ-irradiation with a dose of $4*10^5$ Gy additional absorption bands appear with maxima at 450, 540 and 680 nm. They can be attributed to Co^{3+} ions arising due to the ionization of Co^{2+} ions present in the as-grown crystal, as it is seen from the absorption curve. Excitation with λ_{ex}=442 nm gives strong luminescence of these ions with a peak at about 650 nm. This behavior suggests that γ-irradiation can dramatically change the emission properties of the crystal.

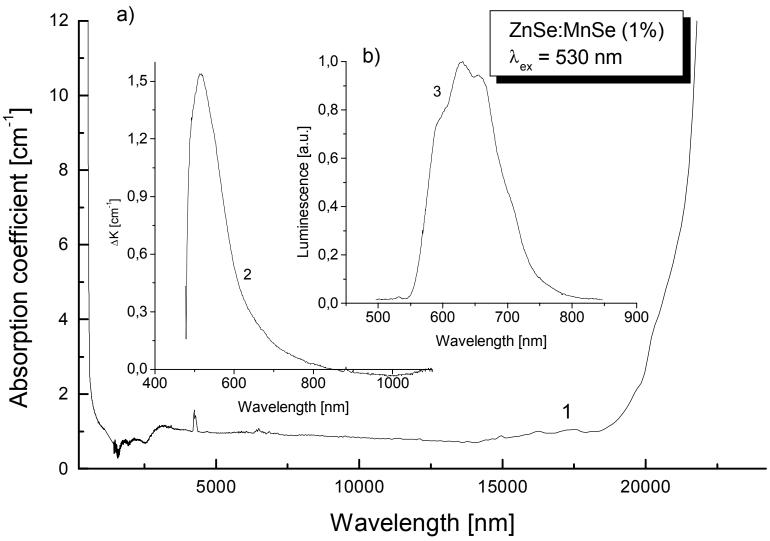

Fig. 8. Absorption (Fig. 8a – curve 1), additional absorption after gamma irradiation with a dose of $4*10^5$ Gy (Fig. 8b – curve 2) and photoluminescence (Fig. 8c – curve 3) of ZnSe: MnSe (1%) single crystal

ZnSe single crystals co-doped with 1% MnSn exhibit similar behavior (Fig. 8). The absorption spectrum of ZnSe: Mn crystal does not exhibit any electron transition bands in the range from 200 to 20000 nm (curve 1). After γ-irradiation with a dose of $4*10^5$ Gy an additional absorption band appears at 520 nm, which is connected probably with Mn^{3+} ions created by gamma ionization of Mn^{2+} ions (curve 2). Luminescence of the crystal with green light (λ_{ex}=530 nm), observed after gamma irradiation, has a maximum at about 650 nm (see Fig. 8b, curve 3).

Transmission measurements have shown that after 1 year of storage of the Nd: YAG single crystal at room temperature the additional absorption after γ-irradiation decreases by 10% [2]. The same measurements performed for Nd: GGG single crystals revealed a fast decrease of additional absorption value in time. ESR measurements revealed that after 1 month of storage of the Nd: SLGO sample at room temperature the intensity of ESR lines generated after irradiation decreases about 10 times which gives the life-time of the observed center of 264 hours [4]. Emission investigations performed for Cr,Tm,Ho: YAG laser have shown that γ-irradiation of a crystal annealed in the air at 1400°C causes stable improvement in the laser emission properties of a Cr,Tm,Ho: YAG crystal with water cooled head [3].

All crystals, which optical characteristics are presented in Figs 6-8, exhibit strong red luminescence at about 650 nm but only in the case of LiF crystals it is due to CC. Other crystals show also new luminescence bands connected with the creation of a new type of active dopants originating from recharged active ions (Co^{3+} and Mn^{3+}).

4. CONCLUSIONS

Generally annealing in the air leads to the increase in valency of uncontrolled or active impurity (Cr^{3+}: YAG), while γ-irradiation (or annealing in reducing atmosphere) (Cr^{4+}: YAG), due to ion recombination with secondary Compton electrons (or reduction), leads to the decrease in valency (Fig. 2) [2]. There are some ions, although which are ionized by gamma quanta (Cr^{3+}: YAG) due to the Compton interaction. It depends on a previous treatment, local symmetry of an ion and crystal field. Protons may also interact similarly as gamma rays. The only difference is in the source of secondary electrons or in ionization mechanism.

Different types of treatments (annealing in reducing or oxidizing atmospheres, irradiation) differ in producing of characteristic defects. They may be CC's, such as F, F^+, F^-, F^{2+}, F^{2-}, polarons, trapped holes [6], AA bands attributed to recharged active (Ce^{3+}, Cr^{3+}, Dy^{3+}, Nd^{3+}) or uncontrolled (Mn^{2+} [10], Fe^{3+}, Fe^{2+}) ions. Effects of gamma irradiation performed for the same crystal but previously annealed in different manner, may be of the opposite sign (see Fig. 3).

If no oxide vacancies are present in a crystal, then AA bands after both types of treatments, i.e. gamma rays and annealing in reducing atmosphere, have the same shape (Fig. 4).

The level of crystal defecting after irradiation depends strongly on its starting quality and also on concentration of active dopant. The quantity of point defects, which may be recharged due to ionizing irradiation, is for the investigated crystals as high as 10^{17} cm^{-3} [2]. Too low annealing can also release new defects (Fig. 5).

For γ-rays with energies ~1.25 MeV recharging processes due to the Compton effect and ionization of active ions were mainly observed.

For electrons with energies <1 MeV (doses of 10^{14}-10^{16} cm^{-2}) ionizing processes of active ions were dominant.

For dose of protons $<10^{14}$ cm^{-2} mainly recombination with delta electrons takes place, while for doses of protons $>10^{14} cm^{-2}$ Frenkel defects also arise, which quantity linearly increases with the dose [4]. This last effect is due to nuclear scattering of protons.

Changes in luminescence spectrum after γ-irradiation were observed for LiF, $Co:LaGaO_3$ and Mn: ZnSe single crystals (Figs 6-8). Such behavior indicates that γ-irradiation can dramatically change emission properties of the crystals.

Recombination processes at room temperature (time constants) which lead to the decrease in additional absorption of the investigated crystals, depend on the type of the crystal.

Thus, irradiation and annealing treatments appear to be the effective tools of crystal change and characterization. Moreover, observed changes in optical properties of some materials after ionizing radiation treatment can have important influence on performance of opto-electronic devices applied e.g. in outer space.

5. ACKNOWLEDGMENTS

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Kontrola stanów ładunkowych: naświetlanie i wygrzewanie w atmosferze utleniającej i redukującej jako metoda zmiany właściwości kryształu i jego charakteryzacji

Streszczenie: W pracy przedstawiono wyniki badań wpływu wygrzewania i naświetlania na właściwości optyczne monokryształów $Y_3Al_5O_{12}$, $YAlO_3$, $SrLaGa_3O_7$, $LiNbO_3$, $Gd_3Ga_5O_{12}$, $LaGaO_3$, ZnSe oraz LiF wykorzystywanych w układach optoelektronicznych (lasery, modulatory). Opisano zmiany walencyjności domieszek

niekontrolowanych (np. Fe^{3+}, Fe^{2+} oraz Mn^{2+}), zmiany walencyjności domieszek aktywnych (e.g. Nd^{3+}, Dy^{3+}, Cr^{4+}, Cr^{3+} and Ce^{3+}), oraz typy centrów barwnych wytwarzanych w kryształach laserowych po ich naświetlaniu kwantami gamma, elektronami i protonami lub wygrzewaniu oraz zmiany zachodzące w widmie luminescencji badanych materiałów.