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STUDY OF LUMINESCENT CHARACTERISTICS OF CHROMIUM-DOPED CRYSTALS

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Abstract

In this article, artificial single crystals $YAlO_3$, $ZnAlO_4$, $MgAl_2O_4$ activated by chromium, and natural crystals of magnesium spinel of the Badakhshan deposit containing 0.03–0.06% chromium were studied. In the emission spectra of these crystals, in addition to emission in the 600 nm band and the R-line, emission in the region of the R′-line was also observed (optical transition $^4A_2 \rightarrow {}^4T_2$), which decreases greatly at 77 K. The maxima of the luminescence excitation bands at 600 nm coincide with the excitation bands of the stationary luminescence of the R-line. In all the studied chromium-activated crystals, antistokes luminescence in the region of 600 nm is observed above room temperature. An increase in temperature to room temperature with the onset of temperature quenching of the U-lines leads to the observation of luminescence of groups of R-lines. With a further increase in temperature, U-glow is detected, located in a shorter wavelength region. A decrease in the glow intensity at higher temperatures indicates the onset of a nonradiative transition of the excited ion to the ground state.

With increasing temperature in YAG-Cr, $ZnAl_2O_4$-Cr and $MgAl_2O_4$-Cr crystals, the quantum yield of U-luminescence, apparently tends to the value of the quantum yield of R-luminescence ($\eta U \rightarrow \eta R$).

Keywords: Crystals $YAlO_3$, Zn_2AlO_4, $MgAl_2O_4$, chromium-doped photoluminescence spectrum, defects.

INTRODUCTION

Luminescence is one of the widely used methods for studying the impurity-defective composition of solids. Therefore, studies of the spatial distribution of luminescent characteristics of doped crystals are of great interest [1]. In [1], samples of zinc selenide doped with chromium were studied. The spatial distribution of the luminescent characteristics of CVD-ZnSe, doped by two-photon confocal microscopy in the spectral range of 0.44–0.73 μm, was investigated chromium using the HIP process. It has been established that as a result of this process, four types of impurity-defective centers are formed in the crystal. It is shown that point centers are involved in their formation, which are formed in the doping zone and diffuse deep into the crystal. Assumptions are made about the nature of these point centers. In [2], a comparative analysis of the features of luminescent properties of three types of ceramic phosphors based on three compounds with a spinel structure: $MgAl_2O_4$, $ZnAl_2O_4$ and $LiAl_5O_8$ activated by manganese and chromium ions was carried out, including temperature dependencies of luminescence spectra. In [3], luminescence spectra of a $YAl_3(BO_3)_4:Cr^{3+}$ crystal were recorded in the spectral range of spin-forbidden electron transitions ${}^2E \rightarrow {}^4A_2$ in Cr^{3+} ions, i.e. in the region of 680–690 nm (14550–14700 cm^{-1}) with high spectral resolution at temperatures of 4–300 K. Temperature dependences of the ratios of integral intensities of the R_2 and R_1 lines, as well as N′ and N (presumably the transition lines ${}^2E \rightarrow {}^4A^2$ of the Cr^{3+} center in a position distorted due to proximity due to some defect) correspond well to the Boltzmann distribution. A radiometric thermometer with absolute sensitivity maximums at temperatures of 40.3 and 21.6 K and a relative sensitivity of up to 12% K^{-1} can be implemented to measure these ratios. Measuring the width of the most intense spectral component, the R_1 - line, can be a way to record temperatures in the range of 100 K and above.

A combination of luminescent thermometry and optical heating was implemented on $YAl_3(BO_3)_4:Cr^{3+}$ (YAB:Cr^{3+}) crystals. Temperature was recorded by measuring the ratio of the intensities of bands ${}^2E \rightarrow {}^4A_2$ and ${}^4T_2 \rightarrow {}^4A_2$ in the temperature range of −150–300 °C [4]. Antistokes luminescence (AL) was observed at 600 nm ions in crystals, YAl Cr^{3+}, ${}^2O_4$, $ZnAlO_3$ and $MgAl_2O_4$. Concentration and temperature dependencies, as well as the relationship between AL and R - luminescence are considered. It is concluded that AL is the result of thermal transfer of an electron from the excited ${}^2E$ state to the ${}^4T_2$ level with a subsequent radiative transition to the main ${}^4A_2$ level. It is shown that AL in the region of 600 nm appears at sufficiently high temperatures (T > 200 K), which depend on the material when excited in all absorption bands at ordinary V, Y, U and anti-Stokes R and R′ excitation. The nature of equilibrium AL due to the specific nature of the configuration curves of C^{r3+} ions is explained in the main and excited states. Theoretically and experimentally, the possible energy levels of chromium ions in ruby, yttrium aluminum garnet (YAG) and magnesium spinel, as well as the absorption and luminescence bands corresponding to many levels from the ground $^4A_2$ and excited ^2E states have been established [5]. However, the issues of configuration curves and the interconnection of these energy levels remain controversial. The study of temperature dependencies of these spectra is important. It is known that with an increase in temperature, the intensity and quantum yield of luminescence of R - lines and the lifetime of level ^2E decrease [6]. Such a decrease cannot be explained by a non-radiative transition $^2E \rightarrow {}^4A_2$, since it weakly perturbs the ambient field. The mechanism of temperature quenching of R-luminescence assumes a thermal transfer of electrons from level ^2E to $^4T_2$. This fully explains the decrease in the lifetime of the ^2E state and the quantum yield in the R-line [6,7]. In the theoretical work of Perlin [6], based on experimental data [7], it is shown that with an increase in temperature, luminescence should be observed in the U region $\rightarrow \eta_u(R) \approx 0,22\ \eta_u(U) \approx 0,19$ For example, in [8], by separating the short-wave part of the spectrum from the R-line of luminescence with a light filter, the band at 700 nm is studied as U- luminescence.

In Cr^{3+}:Al_2O_3 (ruby) crystals with an insignificant concentration of Cr^{3+} impurity (10^{-6} wt.%), narrow spectral R-lines of cathodluminescence (CL) Cr^{3+} are well detected under high-current nanosecond electron excitation [9,10]. For Al_2O_3 crystals containing 10^{-1} wt. % Cr^{3+}, a high intensity of CL of R-lines Cr^{3+} is recorded [9], the high spectral resolution of the instrument is achievable, and is of considerable interest related to the determination of the effect of an induced hypersonic pulse on the spectral parameters of the R-lines of the Cr^{3+} sapphire CL during electron bombardment. The fluorescence spectrum of the YAG sample: 0.05% Cr at room temperature is similar to that of alexandrite, with both $^4T_2$, ^2E – $^4A_2$. The transition contributes to the Cr^{3+} spectra, and a slightly larger energy distance between them (Δ=1000 cm^{-1}) [11] leads to a relatively smaller contribution of the $^4T_2$ – $^4A_2$ transitions to luminescence. The fluorescence of some dielectric crystals activated by Cr^{3+} ions under excitation conditions in the long-wavelength tail of the absorption spectrum is investigated [12]. Cr showed [11] that the tail of the U-band of luminescence is located in this region, and its maximum is located at 600 nm. In the literature, it is believed that during the cascade transition of $^2E \rightarrow {}^4T_2 \rightarrow {}^4A_2$, the transition to the main level of $^4T_2 \rightarrow {}^4A_2$ occurs mainly in a non-radiative way.

The results show that the transition from the excited $^4T_2$ state to the main state. The $^4A_2$ level occurs with radiation in the λ_u absorption region $\leq \lambda_u$ the luminescence $< \lambda_R$. From room temperature onwards, there is a gradual decrease in the quantum yield of R-lines both when excited on the R(η_U) U-band and when R(η_R) is excitation in resonance. At the same temperatures, the lifetime of level ^2E decreases. The reason for this is the thermal colonization of the $^4T_{2g}$ level ($^2Eg \rightarrow {}^4N_{2g}$), which is confirmed by the appearance of luminescence in the U-band with a maximum quantum yield of η_R(R) $\cong$ of 0.22 at 400 K [6]. Energy distance from the ^2E level to the maximum U-band (550 nm) and up to a 597 nm phonon-free line is 0.46 and 0.30 eV, respectively. In [13], attention is drawn to the fact that the beginning of temperature quenching has an activation energy of ~0.08 eV, which corresponds to the transition $^2Eg \rightarrow {}^2T_{1g}$. At high excitation densities, a multistructural luminescence band was found in this region, part of which also binds to U-luminescence. Luminescent spectra of the ceramics show two emission peaks, a strong peak at 872 nm and a weak peak at 960 nm because of $^4T_2 \rightarrow {}^4A_2$ transition of the Cr^{3+} ions. Intensity of the emission peaks increases with Cr^{3+} concentration, reaches maximum with 0.1 at.% Cr^{3+}, then decreases with higher Cr^{3+} concentration. Owing to the differences in crystal field strength, the luminescent properties of the Cr^{3+}: CMS fluorescent ceramics and powder are quite different [14].

The luminescent properties of Cr-doped alumina are well known. The luminescence mechanisms consists of three stages; excitation, non-radiative relaxation and radiative relaxation, which have been illustrated in Figure 1. The excitation can be stimulated by absorption of electromagnetic radiation. Cr-doped -Al_2O_3 shows two broad absorption bands with peak positions in the ultraviolet and green light at around 400 nm and 553 nm, respectively [15]

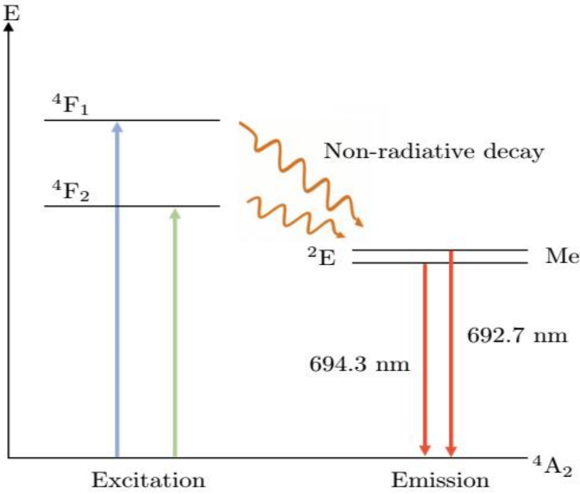

Figure 1: An energy level diagram showing luminescence mechanisms of Cr-doped -Al_2O_3

After an electron is excited from the ground state to a higher energy state, it decays nonradiatively to the intermediate metastable state ^2E, where it further relaxes to the ground state $^4A_2$. This radiative $^2E \rightarrow {}^4A_2$ transition is characteristic of ruby and forms two sharp emission peaks, so-called R-lines. At room temperature, the first R-line is at 692.9 nm and the second at 694.3 nm, and the luminescent lifetime is 3 ms [16], [17]. It is worth mentioning that ESS desires a luminescent material with a shorter luminescent lifetime than 3 ms. The pulses of the proton beam will be 2.8 ms long, which makes 3 ms long luminescence lifetime very long in relation to the duration of the pulse. Studies have shown that the luminescence intensity of the Cr-doped -alumina is significantly lower than ruby [18-28].

In this work, the U-luminescence of chromium ions in other bases with different parameters was studied in order to show the role of the radiative and non-radiative relaxation channels of the excited ion at an increase in temperature.

OBJECT AND METHODS OF RESEARCH

To achieve this goal, artificial single crystals $YAlO_3$, $ZnAlO_4$, $MgAl_2O_4$, activated by chromium, and natural crystals of magnesium spinel of the Badakhshan deposit, containing 0.03–0.06% of chromium, were chosen. In all these crystals, chromium ions replace aluminum, but different local environments change the position of energy levels and the corresponding absorption and luminescence bands.

For the experiments, monocrystalline samples with dimensions of 8x8x3 mm^3 ($ZnAl_2O_4$ -Cr crystals, sizes 3x3x2 mm^3) were made. The excitation and luminescence spectra were studied using the MRG-2A spectral fluorometer (HITACHI). It should be noted that due to the low power of the excitation source, nonequilibrium luminescence and nonlinear effects are not observed in our experiments.

RESULTS AND DISCUSSION

As the results of the studies showed, for various chromium-activated crystals, the temperature dependence of luminescence in the 600 nm region is similar to the results obtained for IAG-Cr [19]. In the luminescence spectra of these crystals, in addition to the luminescence in the 600 nm band and the R-line, luminescence in the R′ -line region was also observed (optical transition $^4A_2 \rightarrow {}^4T_2$), which decreases greatly at 77 K. The maxima of the excitation bands of the luminescence of 600 nm coincide with the excitation bands of the stationary luminescence of the R-line.

Luminescence in $ZnAl_2O_4$-Cr excited in the Y band at different temperatures is shown in Fig.2. The structure, ratio of line intensities and temperature dependence of luminescence of artificial and natural $MgAl_2O_4$-Cr crystals are similar to the luminescence of $ZnAl_2O_4$-C$_r$.

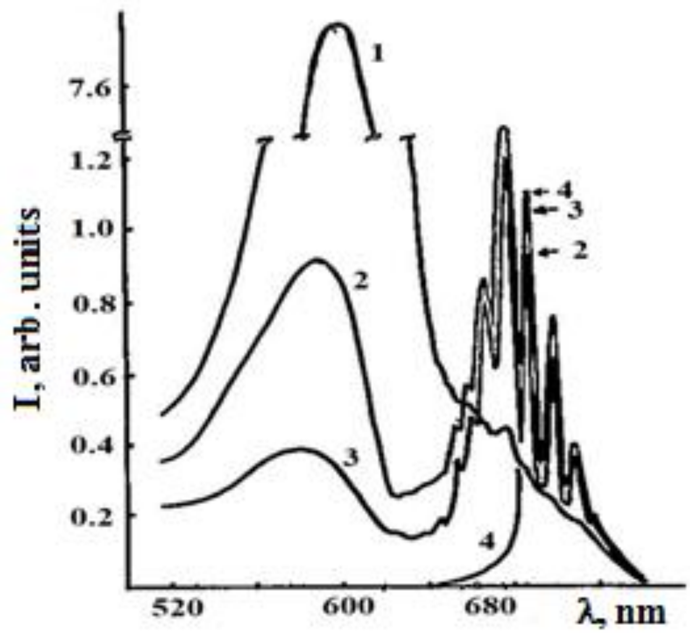

Fig.2. Photoluminescence spectrum of $ZnAl_2O_4$-C$_r$ when excited on the Y-band of absorption at temperatures of 530 K (I), 380 K (2), 350 K (3), 77 K (4)

As can be seen from the temperature dependence of the intensity of U, R′- and R-luminescences (Figure 3), the luminescence in the U-band does not reach saturation at the studied temperatures, from which it follows that in the configuration curves of the studied crystals, the intersection of the basic level ${}^4A_2$ with the excited ${}^4T_2$ level occurs at higher energies than in a ruby. Such a shift can occur when the shape of the configuration curves of Cr^{3+} ions changes, respectively, for each crystal, or when the configuration minimums ${}^4T_2$ and ${}^4A_2$ coincide. Apparently, in $Y_3Al_5O_{12}$-Cr, the coincidence of the absorption band and luminescence (${}^4A_2 \Leftrightarrow {}^4T_2$) speaks in favor of the latter assumption.

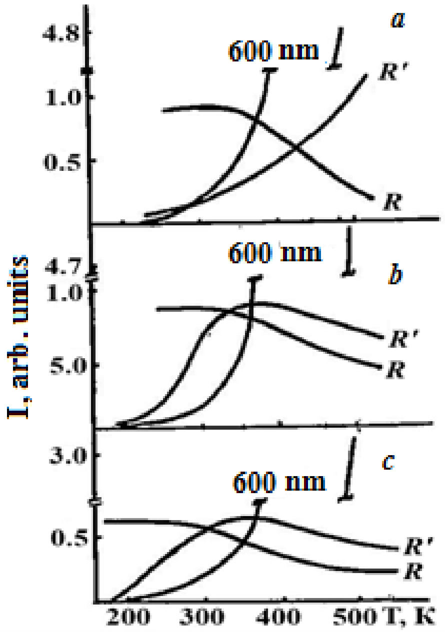

Fig.3. Temperature change in the intensity of the 600 nm luminescence band when $YAlO_3$-Tu,Cr (a), $MgAl_2O_4$-Cr (b), $ZnAl_2O_4$-Cr (c) crystals are excited on the Y-band of absorption

As might be expected, the temperature dependences of the intensity of the R-line and the 600 nm band differ markedly for different types of crystals (Figure 3), since the spectroscopic values of the activation energy of $^4T_2 - {}^2E$ are different for different crystals [20]. Attempts will find some kind of relationship between the activation energies of $^4T_2 - {}^2E$. The existence of the ^2T_I level facilitates the thermal transition from the metastable ^2E state to the $^4T_2$ level, being a step between these transitions $^2E \rightarrow {}^4T_1 \rightarrow {}^4T_2 \rightarrow {}^4A_1$. Luminescence is sufficient to excite chromium ions to level ^2E at high temperatures.

To confirm this mechanism, anti-Stokes excitation spectra were taken in the temperature range of 450÷510 K. In the spectra of anti-Stokes excitation of chromium ions in all matrices, excitation lines corresponding to R- and R′- luminescence were found (Figure 4).

The temperature dependence of the intensity and the shape of the U-band of luminescence does not change during excitation in the V-, Y-, and U- bands, as well as during anti-Stokes excitation in the R- and R′-lines.

It follows from Figures 2 and 3 that the luminescence intensity in the U-band exceeds the maximum intensity of the R-line at appropriate temperatures. The factors leading to this dependence may be as follows:

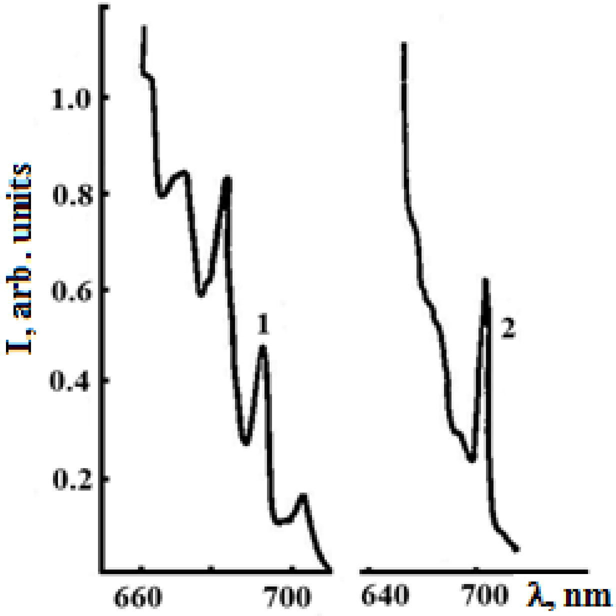

Fig.4. Spectrum of anti-Stokes excitation of the luminescence band at 600 nm in $ZnAl_2O_4$ -Cr (1) and Al_2O_3-Cr (2) crystals at 510 K

I. As the temperature increases, the integral absorption in the excitation bands increases. In particular, in yttrium aluminate and garnet, an increase in integral absorption was observed at an increase in temperature from 77 to 700 K [20]. For ruby crystals, this is excluded [21].

2. In stationary excitation, the intensity of the R - line of luminescence is underestimated due to strong self-absorption.

3. Apparently, an increase in the quantum yield of U- luminescence with an increase in temperature also plays a significant role.

At temperatures when the non-radiative transition of $^4T_2 \rightarrow {}^4A_2$ has not yet occurred, the following ratio can be used approximately to determine the quantum yield of U- luminescence:

$$\eta_U = \frac{I_U \eta_R}{I_R},$$

where ηR, IR and ηU, IU are the quantum outputs and luminescence intensities of the R- line and U- band, respectively. The quantum yield of U- luminescence determined by this formula does not give a satisfactory result, due to the above assumptions. It follows from Fig.3 that already at 350–400 K, taking into account the intensity of the R'-line, the quantum yield is U- luminescence for different crystals is in the range of $\eta=0.3\div0.5$, which indicates a decrease in these values in [6]. Due to the significant yield of luminescence of the U- bands, it can also be seen in the spectra of high-temperature (above 350 K) thermoluminescence of the studied gamma-irradiated samples.

In the excited ion Cr^{3+} in all the studied matrices, all the overlying levels and sublevels in relation to the sublevel corresponding to the R_1 luminescence line, up to the $^4T_2$ level, are in thermal equilibrium with an increase in temperature to 77 K with a decrease in the intensity of U-luminescence in the line, an increase in the intensity of the R-line is observed [22]. An increase in temperature to room temperature with the onset of temperature quenching in the U-lines leads to the observation of the luminescence of groups of R-lines - a glow located in a shorter wavelength region. A decrease in the intensity of luminescence at higher temperatures indicates the onset of a non-radiative transition of the excited ion to the ground state.

CONCLUSION

1. In all the studied chromium-activated crystals, anti-Stokes luminescence in the region of 600 nm is observed above room temperature.
2. The anti-Stokes shift is explained on the basis of the summation of the energy of photons and phonons of the corresponding frequency during the excitation process.
3. The energy transfer between the excited activator and the matrix ions is different for different samples, as evidenced by the temperature change in the yield and the quenching of the U-luminescence.
4. With an increase in temperature in the YAG-Cr, $ZnAl_2O_4$-C_r and $MgAl_2O_4$-Cr crystals, the quantum yield of U-luminescence seems to tend to the value of the quantum yield of R-luminescence (U $\eta_R \rightarrow \eta$).

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