Spectral and kinetic characteristics of the luminescence center in LiF-WO₃ and ZnWO₄ crystals

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Abstract The results of the comparative analysis of the spectral and kinetic characteristics of the luminescence centers in the non-doped ZnWO₄ crystals and in crystals LiF doped with tungsten oxide. It was found that the intrinsic characteristics of the scintillation transition at 2.6–2.7 eV in these crystals do not depend on the energy and symmetry of the crystal lattice, have comparable values of the light output and can be attributed to the luminescence centers of the same impurity nature. The crystal lattice determines the efficiency of the creation and accumulation of radiation defects, presence of which leads to a distortion of the spectral and kinetic characteristics of scintillation.

1. Introduction
Scintillator materials based on oxygen compounds are currently widely used as laser materials, as radiation detectors in medical diagnosis and customs control. Self-radioactivity of these materials is low, the light yield is high, and they are nonhygroscopic and chemically inert. Since the density of these materials is high, they can be used as detectors to conduct research in high energy physics.

Scintillating materials based on oxygen compounds may be divided into two types: scintillators of a doped type and natural scintillators. The radiative characteristics of scintillators of a doped type are determined by the type of the ion-dopant (occupancy of its electron shells). They can be independent of the local symmetry of the dopant position, the type and energetics of the crystal lattice (e.g. intracenter transition in Ce ion in Y₂SiO₅, LuBO, and LiLuSiO₄). The role of the matrix is to transfer energy to the dopant.

In natural scintillators (e.g. Y₂O₃, BeO, tungstates and molybdates of Group II metals [1]) the luminescence centers are matrix forming ions or anionic complexes, the properties of emission transitions in which depend on the symmetry and energy parameters of the crystal lattice.

In this paper, the objects under study were oxygen-containing ZnWO₄ crystals and LiF crystals doped with tungsten oxide (LiF-WO₃). According to [1-6], ZnWO₄ refers to natural scintillator and the luminescence excited in it at 2.6 eV is considered to be intrinsic, caused by self-trapped excitons in oxyanion WO₆ complex as a component of the crystal lattice. In unbroken wolframite lattice, each W⁶⁺ ion is in the center of an asymmetric octahedron formed from six O₂⁻ oxygen ions due to three different W-O distances (1.79, 1.91 and 2.13 Å).

LiF crystal is the scintillator of a doped type. It luminesces when being doped with tungsten oxide, in particular. Therefore, in both crystals, the radiative complex includes a tungsten ion and an oxygen ion. The comparative analysis of the luminescence parameters of the two crystals will help to understand the structure of the luminescence centers, to find out the effect of the lattice type on emission characteristics, to determine the range of doses of ionizing radiation to use the crystals as detectors of ionizing radiation.

2. Object and Methods
The investigated crystals were grown at the Institute for Scintillation Materials, NAS of Ukraine (Kharkiv). LiF crystal doped with tungsten oxide was grown by automated Kyropoulos method in nitrogen atmosphere in the form of a boule with a diameter of 240 mm and a height of 400 mm. The concentration of tungsten measured spectrophotometrically was varied from top to bottom of the boule.
in the range of 0.01–0.5 wt%. ZnWO₄ crystals were grown by Czochralski method from a mixture prepared by solid-phase synthesis of ZnO oxides (99.995%) and WO₃ oxides (99.995%). The initial imperfection of the crystals was evaluated by the absorption spectra in the range of 13–0.1 eV. In the range (6–13 eV), absorption spectra were measured in STIMS SC "S.I. Vavilov State Optical Institute" (St. Petersburg) with a vacuum monochromator-based spectrometer at 300 K. In the VUV region, the absorption spectra of LiF crystals doped with WO₃ contained bands at 6.2, 7.3, 8.9, 11.2 eV. The same spectrum could be observed in LiF-O crystals (figure 1), in which these bands are attributed to transitions in oxygen ions in different complexes: OH⁻, O²⁻, O₂⁻, O²⁻-V₉ [7–11]. The intensity of these bands in LiF-WO₃ crystals increases as the amount of the doped oxide increases. At a concentration of more than 0.2 W wt%, the crystals were not transparent above 5 eV.

The non-transparency of as-grown ZnWO₄ crystals in the region of E> 4 eV (figure 1) is assumed [2–6] to be due to intrinsic absorption. However, this assumption does not agree with the evaluation of the band gap from 4.6 eV to 5.8 according to various authors [2,12,13]. This is one of the reasons why the authors of an alternative point of view [14–16] attribute the non-transparency of ZnWO₄ crystals above 4 eV to high levels of initial imperfection of the material and presence of impurity absorption states at the edge of the band gap.

In the range (0.5-0.1) eV, the absorption spectra were measured in Institute for Scintillation Materials (Kharkov) on a Fourier spectrometer "Spectrum One". IR absorption spectrum of crystals LiF-WO₃ contains two bands at 3728 and 3620 cm⁻¹ caused by free ions OH⁻ and complexes Me-nOH respectively. The appearance of a proton (H⁺) as an interstitial probably is due to the presence of moisture in the atmosphere of growth.

Scintillation properties of crystals (pulsed photoluminescence (PL) and cathodoluminescence (CL)) were investigated using a measurement technique with nanosecond time resolution in the range of 10⁻⁸–10⁻¹ s after the end of pulsed excitation. The intensity of the luminescence was detected with monochromator MDR-206 and photomultiplier PEM-97. The spectra were recalculated for PMT sensitivity.

Pulsed CL was excited by electron pulse with the parameters: pulse duration of 10 ns; the average electron energy of 250 keV. The dose absorbed by the crystal for the duration of a single electron pulse was equal to 5.10¹¹ Gy.

Pulsed PL was studied under excitation by the fourth harmonic of Nd-GNY (4.66 eV) laser with a pulse duration of 5 ns and an energy of 40 mJ per pulse. The excitation spectra of the luminescence at 2.6 eV were measured in a steady state in the range of the photon energy of 3–6.2 eV with
spectrofluorimeter CM-2203. The luminescent properties of the crystals were studied before irradiation and after irradiation by electron beams in the range of absorbed doses of 0–10^5 Gy and temperature range of 15–300 K. To clarify the effect of the surface on the luminescence we analyzed the emission spectra of crystals of different prehistory: crystals freshly cleaved from single block and those stored for 10 hours at 900 C in oxygen atmosphere.

3. Results and discussion
In crystals of both types (LiF-WO_3 and ZnWO_4) the luminescence is excited by both helium-neon laser (4.66 eV), and electron beams. We have established that the parameter of the luminescence decay depends on the radiation wavelength. Figure 2 shows an example of the kinetics of the luminescence decay for ZnWO_4 crystal. A short decay component only can be clearly seen in the UV region of the spectrum. Its contribution decreases as the wavelength of the emitted energy grows, and the contribution of the long component decay increases. In the region of energies <2.95 eV (to the right), a short luminescence decay component is not observed in the decay kinetics. The spectra of the decay components in crystals of both types (ZnWO_4 and LiF-WO_3) are a band at 3.6 eV with the decay parameter of \(\tau<20\) ns at 300 K, and the band in the region of 2.6–2.7 eV with decay parameter of 30 \(\mu\)s at 300 K. Figure 3 shows an example of the spectrum of pulsed PL crystal for ZnWO_4.

The results obtained in the study of the properties of the bands in the luminescence spectra for both crystals are as follows.

• The short-lived band at 3.6 eV is present in all ZnWO_4 crystals, and its intensity increases during crystal storage in air at 300 K, and when heated at 900 C in oxygen atmosphere.

As we showed in [17] the band at 3.6 eV is not found in undoped LiF crystals grown by Stockbarger in fluorinating atmosphere, and it is observed in all LiF crystals doped with different oxides: Li_2O, WO_3, TiO_2, and Fe_2O_3. Neither the spectral position of this band nor the lifetime (<20 ns) of the corresponding luminescence center depends on the type of cation-co-dopant.

• The ratio of the intensities of the bands at 3.6 and 2.6 eV (J(3.6)/J(2.6)) in both LiF-WO_3 and ZnWO_4 crystals depends on the conditions of luminescence excitation. In ZnWO_4 crystal this ratio is two orders of magnitude greater under excitation of the luminescence by photon flux with the energy of 4.66 eV than under excitation of luminescence in the same crystal by electron beams. The penetration depth of 4.66 eV photons in the crystal is less than 100 nm, while the depth of electron penetration into the crystal is equal to 200 \(\mu\)m. Therefore, the reduction of the ratio (J(3.6)/J(2.6)) with the increase of the excitation radiation penetration depth in the same crystal indicates that centers
responsible for the band at 3.6 eV are preferably located in the surface layer of the crystal. The fact that the band at 3.6 eV increases in heating the crystals at 900 C for 10 hours discovered by us indicates that the luminescence centers responsible for this band are not intrinsic defects of the crystal. The second band in the luminescence spectrum of LiF-WO$_3$ and ZnWO$_4$ crystals is the band at 2.6–2.7 eV. Its properties are as follows.

• The light yield in intensity of the long-lived band at 2.7 eV increases as the concentration of tungsten in LiF crystals grows (figure 4) and at concentration W equal to 0.32 wt % is 18% with respect to the light yield of CdWO crystal.

• For LiF crystals doped with different MeO oxides we showed [17] that the position of long-lived luminescence band at 3–2 eV and the value of the longest component of the luminescence decay at 300 K depend on the type of metal ions in the oxide. Table 1 shows the results indicating the shift in the long wavelength region of the maximum position of the dopant band from 3 to 2.7 eV (by 10%) and decrease in the decay constant at 300 K from 400 to 30 µs (by 92%) in series Me: Li, Fe, Ti and W. These results irrefutably testify that the parameters of the emission transition in LiF crystals doped with oxides of different metals are affected by the cation co-dopant, which is in the vicinity of the oxygen ion. Increase in the probability of emission transition, as the atomic weight of the cation-dopant increases, indicates the so-called heavy-atom effect, first observed for organic molecules in [18]. In our case, we assume that under excitation, the wave functions of oxygen and metal

### Table 1. Spectral and kinetic parameters of the luminescence at 300 K for ZnWO$_4$ and doped LiF crystals.

| Crystal     | Type of dopant | Band position (eV) | Component of emission decay (ns) | Band position (eV) | Components of the emission decay (µs) |
|-------------|----------------|--------------------|----------------------------------|--------------------|--------------------------------------|
| ZnWO$_4$    | -              | 3.6                | <20                              | 2.65               | 0.2; 3; 30                           |
| LiF         | Li$_2$O        | 3.6                | <20                              | 3.6                | 400                                  |
|             | Fe$_2$O$_3$    | 3.6                | <20                              | 3.2                | 180                                  |
|             | TiO$_2$        | 3.6                | <20                              | 3                  | 120                                  |
|             | WO$_3$         | 3.6                | <20                              | 2.7                | 0.2; 3; 25                           |

Figure 3. Absorption (1) and CL spectra (2,3) for ZnWO$_4$ crystal measured at 300 K with a delay of 10 ns (2) and 50 µs (3) after the end of excitation by electron beam. CL spectra are normalized.
overlapping, and the electron is transferred from excited oxygen ions \((\text{O}^{2-})^*\) to metal ion \(\text{Me}^{n+}\) to form an unstable impurity complex \(\text{Me}^{(n-1)+} - \text{O}^{-}\), which lifetime is entirely determined by Me ion. The existence of a correlation between the value of the light yield and the intensity of an absorption band at 3620 cm\(^{-1}\) in the spectrum of activated crystals with increasing of tungsten concentration in the crystal (figure 4) does not contradict with this assumption.

• The charge state of the dopant ion changes from +1 (Li) to +6 (W) and determines the number of coordinated \(\text{O}^{2-}\) ions, as well as the type and amount of cation or anion vacancies ensuring local compensation of the excess charge. (For example, in substituting the Li\(^+\) ion by W\(^6+\) ion, the excess charge can be compensated in different ways: in form of 5O\(^2-\) or \((4\text{O}^{2-} + \text{V}_c)\) or \((6\text{O}^{2-} + \text{V}_a)\) and etc.

Weak dependence of the emission energy in Me-O complex on the charge state of Me (shift at 0.3 eV in series from Li\(^+\) to W\(^6+\) (table 1) indicates insignificant impact of the point symmetry of the center in the lattice on the value of transition.

\[ \text{Figure 4. Dependence of absorption coefficient at 3620 cm}^{-1} \]

and the relative light yield at 300 K in the band at 2.7 eV on the concentration of W in LiF-WO\(_3\) crystals

• To find out the nature of the luminescence band at 2.6 eV in the ZnWO\(_4\) crystal we studied the spectrum of its excitation by photons in the energy range of 3–6.5 eV. The obtained data presented in figure 5 indicate: 1) the existence of the excitation energy threshold equal to 4.1 eV; 2) insignificant (10%) decrease in the band intensity as the photon energy grows in the range of 4–6.5 eV; 3) decrease in the intensity of the band at 2.6 eV as the energy of exciting photons exceeds the threshold in crystals subjected to heating at 900 C for 10 hours (by 80% for the photon energy equal to 6.5 eV).

The results show that the threshold energy (4.1 eV) to excite the band at 2.6 eV in ZnWO\(_4\) does not coincide with the estimated value of the transparency cutoff 4.6–5.8 eV, as reported in [2,12,13]. This fact in [19] is attributed to the ability to create self-trapped excitons in WO\(_6\) complexes responsible for the luminescence in the band at 2.6 eV if the photon energy falls into the Urbach "tail" on the edge of the transparency cutoff.

However, the mismatching of value of the threshold energy (4.1 eV) to excite the band at 2.6 eV with position of edge of intrinsic absorption can be considered as direct proof of the impurity nature of the luminescence at 2.6 eV excited in the impurity absorption region in the transparency cutoff. (Maintaining the stoichiometry of the melt during crystal growth is known to be difficult [20]).

Decrease in the efficiency of excitation of the band at 2.6 eV, as the photon energy increases in the heat-treated crystals (figure 5), directly indicates a change in the properties of the crystal surface during heating and indicates sufficiently high chemical activity of the material.

To determine the effect of the lattice type on the properties of emission centers, we conducted a comparative analysis of characteristics of the radiative transition in the region of 2.6 eV in the tested crystals. We obtained the following results.
In ZnWO₄ crystal at 300 K, FWHM of the band at 2.6 eV is equal to 0.6 eV, and the luminescence decay is described by a set of three exponential functions with values of decay constants of 30 µs, 3 µs and 200 ns at 300K. In LiF-WO₃ crystals: the band is found at 2.7 eV, and the decay is described as a set of three exponential functions with values of decay constants of 200 ns, and 3 and 30 µs at 300 K similar to that in LiF-WO₃. The spectra of all the decay components are identical. They represent are a band with a maximum at 2.6 and 2.7 eV in ZnWO₄ and LiF-WO₃, respectively.

The results obtained show that as temperature increases in the range of 15–250 K in LiF-WO₃ crystals, the intensity of the band at 2.7 eV increases, and the duration of the decay reduces by an order of magnitude from 230 to 35 µs (figure 6). According to [21], in ZnWO₄ crystals, increase in the intensity and decrease in the decay time from 160 to 35 K occur in a narrow temperature range of 5–15 K. In the region of 15–250 K, the luminescence parameters remain virtually unchanged.

The light yield in the band at 2.6 eV at a temperature range of 15–250 K does not depend on the temperature in crystals of both types, but it is achieved in different ways: in ZnWO₄ crystal both J=const and τ=const in this temperature range, whereas in LiF-WO₃ crystal J=f(T), and it increases as the temperature of the crystal grows under excitation; τ=f(T), and it decreases as the temperature rises. However, the result of multiplying Jτ remains constant and independent of temperature.

Thus, the only distinguishing feature of the luminescence in the region of 2.6–2.7 eV in ZnWO₄ and LiF-WO₃ crystals is the difference in the temperature range of change in decay parameters: 4–15 K (in ZnWO₄) and 15–250 K (in LiF-WO₃ crystals). Within the approximation of an adiabatic potential emission center in the ground and excited states as a functions of the generalized configuration.

Figure 5. Spectra of luminescence excitation at 2.6 eV in different ZnWO₄ crystals: freshly cleaved (1) and heated at 900 C (2).

Figure 6. Temperature dependence of the decay time (1) and the intensity of the CL at 2.7 eV (2) in LiF-0.2 wt % W.
coordinate it implies different barrier height for transition of the emission center from an excited state to the ground state in these crystals. This can be due to either different degree of lattice relaxation in the luminescence center after its transition to an excited state or significant change in the elastic constants of the lattice for the center in an excited state. This is to be investigated.

The light yield in the long-lived luminescence band in the spectrum of ZnWO$_4$ crystal is 76% relative to CdWO$_4$ crystal [20]. According to our results the light yield in the LiF-0.32 wt % W is 18% relative to a reference CdWO$_4$ crystal. This means that the light yields in the bands at 2.6 (ZnWO$_4$) and 2.7 eV (LiF-WO$_3$) are of the same order of magnitude. The latter fact contradicts the assumption about the intrinsic character of the luminescence in the band at 2.6 eV in ZnWO$_4$ crystal since the number of a matrix-forming emission W-O complexes per unit of the volume is at least four orders of magnitude greater than activator centers in LiF-WO$_3$ crystal (~10$^{17}$ cm$^{-3}$).

Thus, the parameters of the emission that is excited optically with photon energy> 4.66 eV and under ionizing radiation for luminescence centers in LiF-WO$_3$ and ZnWO$_4$ crystals are comparable. We studied the range of the absorbed doses within which the emission characteristics of the crystals remain constant.

We have established the following. In LiF-WO$_3$ crystals at 300 K under electron beams irradiation the luminescence parameters are independent of the absorbed dose in a limited range of 10$^1$–10$^3$ Gy. Further increase of the dose is followed by degradation of spectral and kinetic parameters of the luminescence, as we described in [22]. The degradation is exhibits itself as a distortion of the luminescence band (figure 7) followed by decrease in the amplitude of the band (figure 7, insert), and as an increase in the afterglow time. The degradation is caused by overlapping of the scintillation band and absorption and luminescence bands of radiation defects accumulated during electron beam irradiation. The failure in the emission band (figure 7) is caused by reabsorption of the emission by F$_2$ color centers. Increase in the afterglow period we attribute to post-radiation interaction of radiation-induced defects [22].

![Figure 7](image_url)

**Figure 7.** The PL spectrum before irradiation (1) and after irradiation by electron beams (D=2.10$^4$ Gy) in LiF-0.31wt % W (2). The insert shows the dose dependence of PL intensity at 2.7 eV at 300 K.

In contrast to LiF crystals, in ZnWO$_4$ crystals, the parameters of the emission at 2.6 eV remain constant in a wide range of the absorbed doses of 10$^1$–10$^5$ Gy at 300 K. In the spectral region of the luminescence we did not observe bands (emission or absorption) due to short-lived radiation defects.
The absence of radiative and absorption transitions in the region of the luminescence band at 2.6 eV in stationary measurements is reported in [14]. It is obvious that these differences in the dose dependence of the luminescence parameters in the investigated crystals are caused by the properties of the crystal lattice that determines the type and efficiency of formation and accumulation of radiation defects.

4. Conclusions

1) Emission transitions at 3.6 and 2.6 eV in both ZnWO₄ and LiF-WO₃ crystals belong to oxygen-containing centers of different types. When the crystal is heated in oxygen atmosphere, the band at 3.6 eV increases due to the destruction (transformation) of the centers responsible for the band at 2.6–2.7 eV.

2) In ZnWO₄ and LiF-WO₃ crystals, the following spectral and kinetic parameters of the emission transition in the region of 2.6–2.7 eV are found to coincide:
- the probability of emission transition at 300 K (table 1);
- the number and value of the components of the luminescence decay at 300 K (table 1);
- independence of the light output on the temperature in range of 15–250 K;
- independence of the light output at emission bands at 2.6 and 2.7 eV on the dose values in the range of 10¹–10³ Gy under irradiation of crystals by electron beams.

The obtained results indicate identical structure of the emission centers in the tested crystals, and show no effect of the crystal lattice and the point symmetry of W-O emission complexes on the properties of the emission transition in the region of 2.6–2.7 eV.

3) In ZnWO₄ crystals, W-O emission complex is not a regular matrix-forming component, and it is located near the lattice defects. The following facts can be used in favor of this hypothesis:
- the absorption in the region of 3.5–4.6 eV (the edge on the transparency cutoff) indicates the presence of defects in the crystal structure of the initial crystal (figure 3);
- the excitation energy threshold (4.1 eV) of the luminescence band at 2.6 eV (figure 5) is found in the region of the impurity absorption, it does not coincide with value of the transparency cutoff crystal and indicates the impurity nature of the excited luminescence;
- the concentration of W-O emission centers in doped LiF-WO₃ crystals does not exceed 10¹⁸ cm⁻³.

Since the light yield in ZnWO₄ and LiF-WO₃ crystals is of the same order, the number of emission centers in ZnWO₄ crystals is four orders of magnitude less than regular lattice sites.

4) The type of the crystal lattice, its energy parameters determine the effectiveness of formation and accumulation of radiation defects in the crystal as the absorbed dose increases. The spectral overlapping of the bands of absorption and emission of radiation defects and the luminescence center makes the emission parameters depend on the value of the absorbed dose, i.e. it leads to degradation of scintillation luminescence (figure 7). The latter, according to our estimates, does not allow use of LiF-WO₃ crystals in hard radiation fields as radiation detectors in a dose range of ≥5 10³ Gy.

In ZnWO₄ crystals, the luminescence parameters remain unchanged in a wide range of the absorbed doses of 10⁻¹⁰⁵ Gy at 300 K. This indicates the possibility of their use as detectors of ionizing radiation in a wide range of doses and temperatures.

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