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G.K. Alpyssova[1], D.A. Afanasyev[1*], Zh.K. Bakiyeva[1], L.A. Lisitsyna[2], M.G. Golkovski[3], A.K. Tussupbekova[1], A.A. Kissabekova[4], S.D. Tuleuov[1]

[1]*Karaganda Buketov University, Karaganda, Kazakhstan;*
[2]*Tomsk State University of Architecture and Building, Tomsk, Russia;*
[3]*Budker Institute of Nuclear Physics of the Siberian Branch of the Russian Academy of Sciences, Novosibirsk, Russia;*
[4]*Pavlodar Pedagogical University named after A. Margulan, Pavlodar, Kazakhstan*
**Corresponding author's e-mail: a_d_afanasyev@mail.ru*

Optical properties of $ZnWO_4$ ceramics obtained by radiation synthesis

One of the promising methods for producing ceramics from tungstate metals of the MWO_4 composition is radiation synthesis using powerful electron fluxes. Due to the unique properties of radiation, it was possible to significantly accelerate the synthesis process. It has been demonstrated that $ZnWO_4$ ceramics can be synthesized by acting on an electron flux with an energy from 1.4 to 2.5 MeV and a high power density of 10–25 kW/cm^2. In this regard, studies of the optical properties of ceramic samples of zinc tungstate $ZnWO_4$ obtained by this method were carried out. The morphology of the surface, X-ray diffraction spectra and optical properties of $ZnWO_4$ ceramics obtained by radiation synthesis were studied. X-ray diffraction measurements and EDX analysis confirmed the formation of zinc tungstate ceramics $ZnWO_4$ as a result of radiation synthesis. The absorption, luminescence, and luminescence excitation spectra of synthesized samples were measured. Luminescence spectra of a reference single crystal sample were also measured. X-ray diffraction confirmed the formation of zinc tungstate ($ZnWO_4$) as a result of radiation synthesis. The luminescent spectra of the synthesized sample coincide with the spectra of crystalline $ZnWO_4$. A comparison of the luminescence spectra of a synthesized ceramic sample and a monocrystalline reference sample measured under different optical excitation conditions shows significant differences in the luminescence spectra of the synthesized sample and the reference sample and indicates possible various defects present in the compared samples.

Keywords: ceramics; luminescence; radiation synthesis; zinc tungstate, X-ray diffraction spectra, EDX analysis, power density, optical properties of ceramics.

Introduction

Most scintillation materials used in ionizing radiation detectors are used in single crystal form. It is known that growing scintillation crystals with high melting point is a labor-intensive and expensive process [1]. One of the promising approaches is the replacement of single crystals by ceramic materials, for example, on the basis of alumina yttrium garnet $Y_3Al_5O_{12}$ (YAG) with metal tungstate ceramics, the study of which has been carried out for quite a long time [2–5]. Due to their unique scintillation properties, such as isotropy, possibility of giving any shapes [2–7], advantages over monocrystalline materials, optical ceramic materials are promising scintillators and detectors and are characterized by high efficiency, mechanical and thermal stability. In contrast to technologies for the creation of single- crystalline materials, ceramic technology offers the possibility of fabricating highly transparent and large- sized samples at lower synthesis temperatures with homogeneous or controlled gradient distribution of activator ions [8–16], which is its undoubted advantage. Nevertheless, today the technological peculiarities of creating ceramic materials are still the subject of active study.

Metal tungstamates of alkaline-earth and transition group metals of the composition MWO_4 (M — Zn, Mg, Ca, Mn, Ca, etc.) with a tungstate structure have long been of practical interest because of their attractive luminescence [17, 18]. $ZnWO_4$ has been applied as a possible new material for microwave amplification by stimulated emission [19], scintillator [20, 21] and optical hole grating material [22], etc. Recently, new applications of this material have emerged, including large-scale bulk scintillators for high-energy physics [23]. In particular, $ZnWO_4$ is a wide bandgap semiconductor with a bandgap width close to 4 eV [24] and is a promising material for new generation radiation detectors.

However, there are still many questions that require further study of these materials, so in a number of studies proved the existence of nanodefects confirmed by studies of luminescence LiF:WO_3, MgF_2:WO_3, metal tungstates [25, 26]. In crystals of tungstate luminescence is caused by the existence of their own lattice defects, in crystals of alkaline-earth metal fluorides luminescence is caused by defects introduced with tungsten [27, 28].

Due to the complex of certain properties, luminescent materials are widely used. As a rule, such substances function under conditions of high external loads and therefore they should be resistant to thermal, mechanical, radiation effects. The most promising is the use of ceramic materials, which accumulate the advantages of crystalline and glassy materials. However, the process of synthesizing ceramics from refractory compounds is rather complex, involves many steps, time- and energy- consuming. Many existing technologies for the synthesis of ceramics from refractory materials, focused on a particular type of compounds, are characterized by low reproducibility. There is a need to develop new synthesis methods that are more universal, efficient and controllable. It is possible to significantly accelerate the synthesis of refractory optical materials by stimulating solid-phase reactions in the radiation field. It can be expected that the inclusion of new effects in the synthesis process, reactions between intermediate products of radiolysis, will not only increase their speed, but also open up new possibilities for the creation of composite materials.

One of the promising methods of synthesis of ceramics with metal tungstate of composition MWO_4 is the method of radiation synthesis in the field of powerful electron fluxes. It is supposed to use a powerful flow of high-energy electrons to affect wide-gap refractory materials. In dielectric materials, the main share of radiation (99 %) is spent on the creation of electronic excitations. At high power density (up to 30 kW/cm^2), high electron energies, more than 1 MeV, high ionization density and high concentration of intermediate products of radiolysis are created in the material volume, which leads to the development of effective diffusion processes, solid-phase and gas-phase reactions in the charge of precursor powders. This feature is supposed to be used in the proposed project for the synthesis of promising luminescent materials for LED's, scintillators, dosimeters. The possibility of synthesis is demonstrated on the example of synthesis of scintillation material based on MgF_2 activated by tungsten, YAG:Ce phosphor ceramics [29].

It is necessary to know their optical properties of scintillation materials for using them. Therefore, the work was carried out to study the optical properties of synthesized ceramic samples of zinc tungstate $ZnWO_4$ synthesized by radiation synthesis.

Experimental

Zinc tungstate ceramics ($ZnWO_4$) were used in this work. These samples were obtained by radiation synthesis from WO_3, ZnO powders at the UNU Stand ELV-6 electron beam accelerator at the G.I. Budker Institute of Nuclear Physics of the Siberian Branch of the Russian Academy of Sciences (G.I. Budker Institute of Nuclear Physics of SB RAS). Electron beams with energies ranging from 1.4 to 2.5 MeV were used. These electron fluxes are sufficient for synthesis of samples with sizes up to 15 cm^3. More details on the technique of sample synthesis can be found in [29]. The single crystal $ZnWO_4$ was also used as a reference standard.

X-ray diffractograms of the samples were obtained using a Bruker D8 ADVANCE diffractometer equipped with a scintillation detector. Sample identification was performed using a database of powder diffraction files, and indexing was performed using EVA software (Bruker, 2007) and using literature data [30].

The surface structure of tungstates was investigated. The study was carried out on a scanning electron microscope (SEM) Mira 3 (TESCAN). To prepare the material for the study, the samples were fixed on a special conductive tape. Since the investigated samples are dielectrics, the samples were coated with a conductive layer of carbon on a Quorum Q150R ES sputtering machine. The study was carried out at an accelerating voltage of 25 kV. Secondary electrons (SE (secondary electrons) detector) and back scattered electrons (BSE, back scattered electrons) detectors were used. The EDX detector, INCAPentaFET-x3, Oxford Instruments, England, was used for EDX analysis.

Absorption, luminescence and luminescence excitation spectra were measured using an AvaSphere 50-HAL-12V integrating sphere, AvaSpec- ULS2048BCL-EVO spectrometer (Avantes, Netherlands) and AvaLight DH-S deuterium-halogen light source were used to measure absorption spectra. Luminescence and photoluminescence excitation spectra were measured on a SM 2203 Solar spectrofluorimeter. Luminescence spectra were also measured using an AvaSpec-ULS2048BCL-EVO spectrometer with two types of excitation sources UV region of mercury lamp emission obtained with UVS6 filter (ultraviolet glass number six) and 355 nm laser AO-355A-1W.

Results and Discussion

Initially, the surface morphology of the synthesized ceramics was studied (Fig. 1). The sample has a heterogeneous structure and there are microparticles on the surface of the sample of various shapes and sizes. These particles may be the particles from which the ceramics were synthesized.

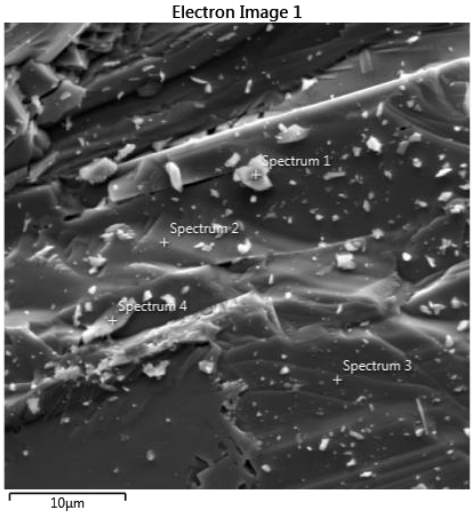


Figure 1. SEM images of the surface of zinc tungstate sample

The EDX spectrum of the sample surface was measured. The EDX spectra confirm the presence of W (1.8, 8.2, 9.5 keV, etc.), Zn (1, 8.5 and 9.5 keV) and O peaks (0.5 keV), Figure 2. The stoichiometric composition of the samples obtained from the measurements is given in Table, where, *G* — a mass concentration of elements, *At* — an atomic concentration of elements, σ — a standard deviation of the measured value. The stoichiometric composition was determined from averaged data.

Table

EDX analysis data of zinc tungstate sample

Element	*G*, %	σ	*At*, %
Zn	18,3	2,12	13,3
W	57,4	2,58	14,8
O	24,3	3,17	71,9

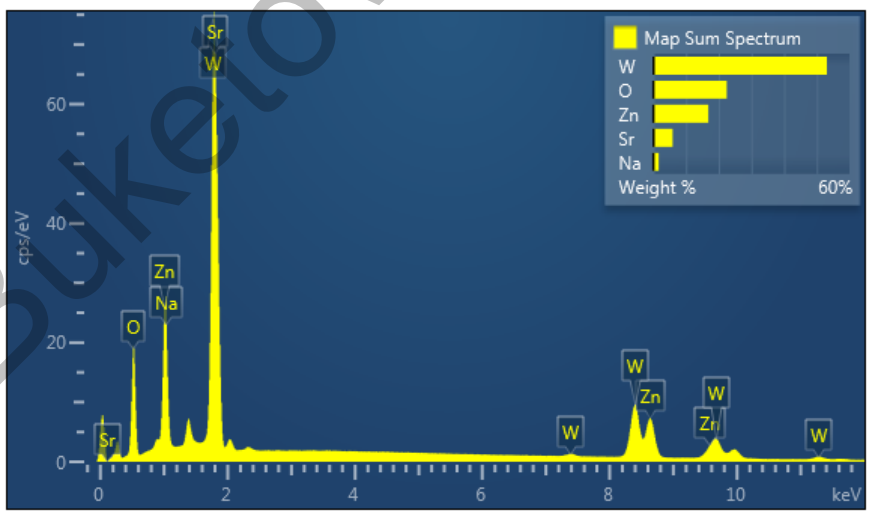


Figure 2. Energy-scattering characteristic X-ray spectra of the sample

According to the atomic concentrations of elements in the sample, the stoichiometric composition of the sample was estimated. It amounted to $ZnW_{1,12}O_{5,43}$. This composition does not significantly differ from the required composition of $ZnWO_4$. The discrepancy can be related both to the accuracy of EDX analysis and possible changes in the composition of ceramics.

The measurement map of the EDX spectra is shown in Figure 3. The elemental composition is shown in Figure 4. Comparison of the data shows that the distribution of tungsten and oxygen is uniform. While zinc

is not evenly distributed on the surface in the microstructure having predominantly green color in Figure 3 zinc content is reduced. In the rest of the map it is distributed quite uniformly. Thus, it can be said that the microstructure with reduced zinc content may be various tungsten oxide compounds WO_2, WO_3 and possibly intermediate oxides $W_{20}O_{56}$ and $W_{18}O_{49}$ [31].

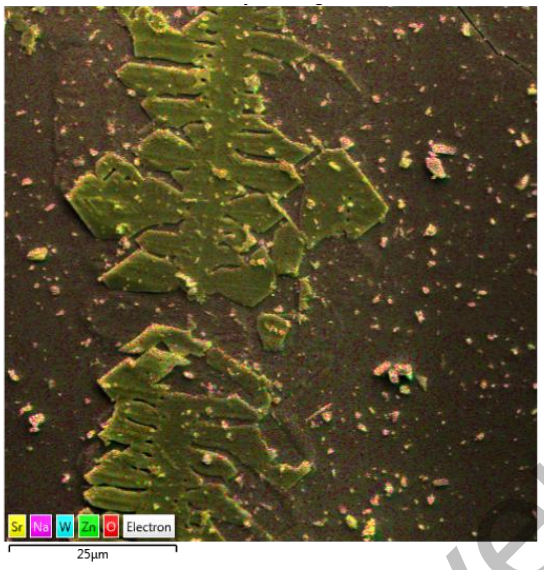


Figure 3. Map of EDX analysis of the sample composition surface

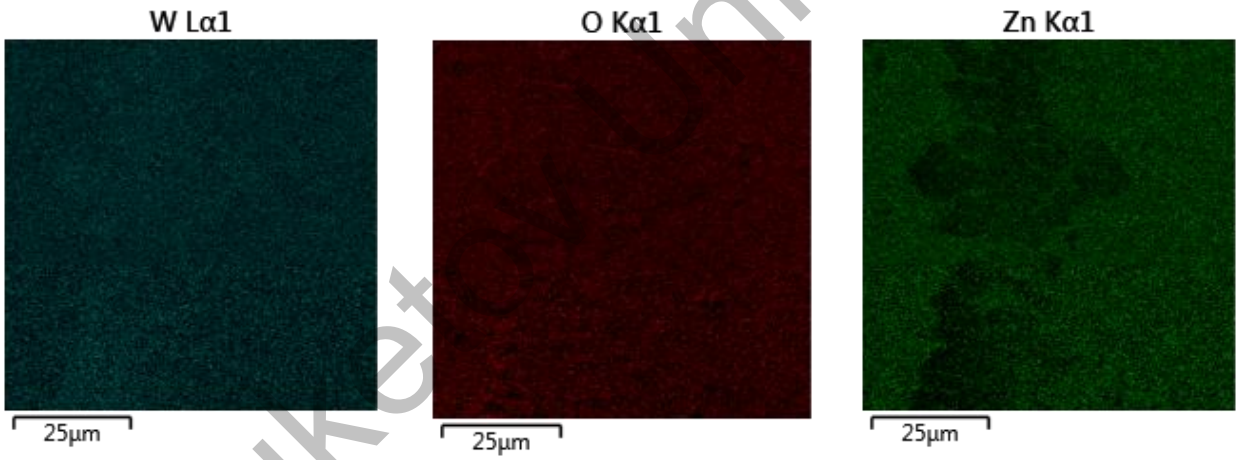


Figure 4. Map of distribution of elements (W, O, Zn) on the surface of the sample

X-ray diffractogram of zinc tungstate $ZnWO_4$ was obtained (Fig. 5).

In Figure 5, all diffraction peaks can be attributed to $ZnWO_4$ using a standard map (JCPDS map No. 73-0554). No peaks from other crystallized phases were observed, which also indicated the formation of $ZnWO_4$ crystals [30].

X-ray diffraction and elemental composition measurements confirmed the formation of zinc tungstate $ZnWO_4$ by radiation synthesis.

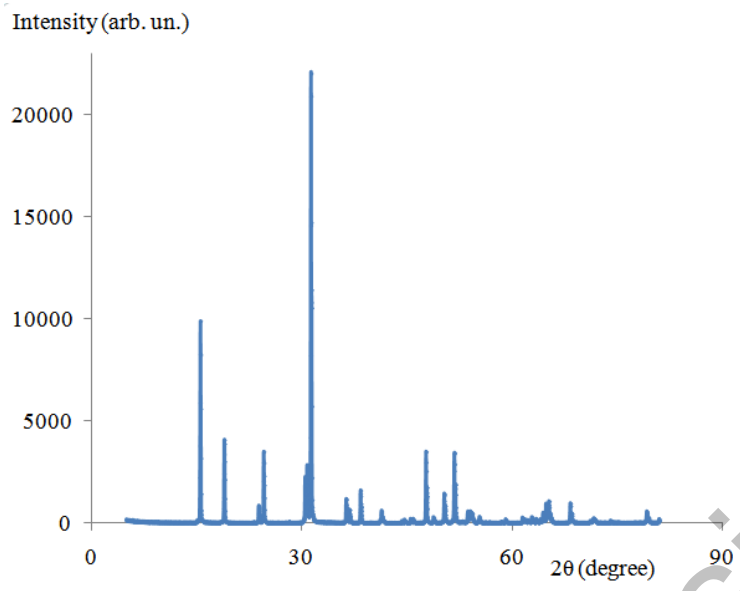


Figure 5. X-ray diffractogram of $ZnWO_4$

Optical properties of the ceramics were investigated. Absorption spectra were measured for the sample (Fig. 6). Since the samples are opaque absorption spectra cannot be measured by standard methods. Therefore, the measurements were performed using an AvaSphere 50-HAL-12V integrating sphere and an AvaSpec-ULS2048BCL-EVO spectrometer. The AvaLight-DH-S compact combined deuterium-halogen light source was used as a light source.

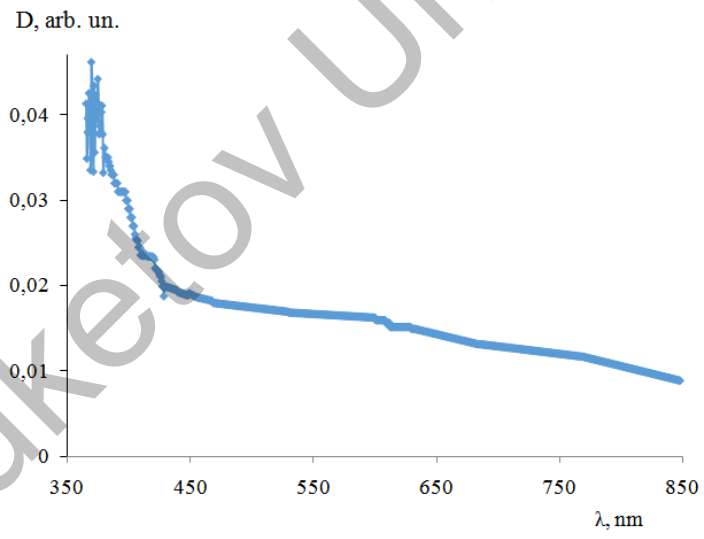


Figure 6. Absorption spectrum of zinc tungstate in the wavelength range from 350 to 1100 nm

Measurement of absorption spectra showed an increase in the optical density of the samples starting from 450 nm to 350 nm. A similar increase in the intensity of the optical density of $ZnWO_4$ samples was also observed in [32]. It is shown that initially observed growth of optical density of $ZnWO_4$ crystal from 420 nm to 340 nm is associated with the presence of impurities such as iron and/or chromium [32], further from 340 nm there is an intense growth of optical density of the crystal associated with the fundamental absorption of $ZnWO_4$ [32, 33].

The absorption spectrum in the range from 200 nm to 350 nm could not be recorded due to the low intensity of the excitation source in this region of the spectrum. For better measurement of absorption spectra in the UV region of the spectrum, a more powerful UV source is needed.

From the absorption spectra, the energy of transitions associated with absorption in the region from 350 to 450 nm was determined. It amounted to 2.25 eV. This value may indicate the possible absorption of de-

fects in synthesized ceramics $ZnWO_4$ since the width of the forbidden zone of the forbidden zone (E_g), according to literature data is 3.98 eV [33].

Luminescence and luminescence excitation spectra were measured. The luminescence excitation and luminescence spectra are shown in Figures 7 and 8, respectively.

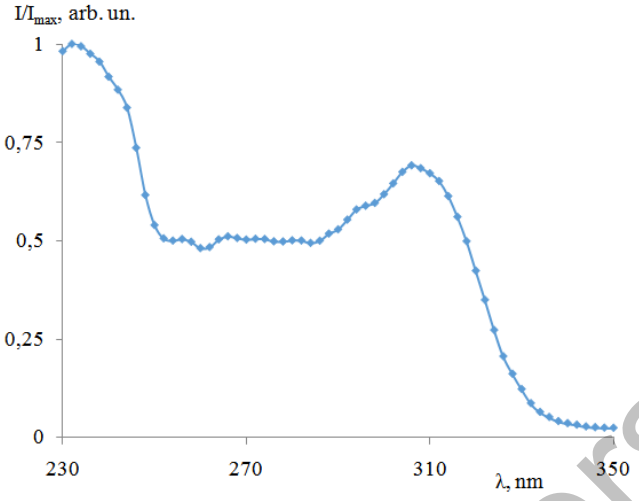


Figure 7. Excitation spectrum of $ZnWO_4$ luminescence with signal detection in the luminescence maximum at 494 nm

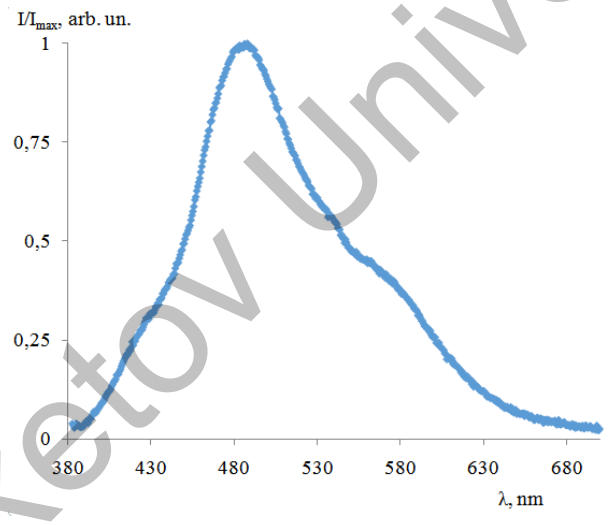


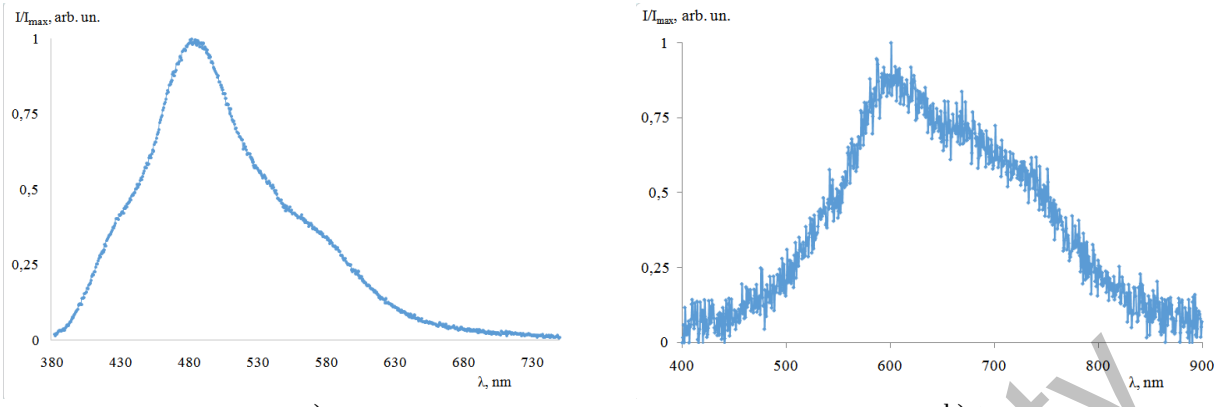
Figure 8. Luminescence spectrum of $ZnWO_4$ under excitation by a mercury lamp at λ_{max} = 490 nm (250–320 nm) (*1*) and spectrum under excitation by a laser source at 355 nm (*2*)

In the excitation spectrum, a band in the region from 200 to 260 nm is observed. A band in the region of 300–320 nm with a maximum at 310 nm is also observed.

Luminescence spectra of the samples obtained at excitation in the region of 250–320 nm under excitation by a mercury lamp have the following characteristics: maximum luminescence intensity λ_{max} = 494 nm, half-width of the luminescence spectrum $\Delta\lambda_{1/2}$ = 46 nm. Luminescence spectra at excitation of samples at 355 nm (AO-355A-1W) shifted to the red region of the spectrum relative to the luminescence spectra of samples excited at 250–320 nm are observed. The maximum luminescence intensity and half-width of the luminescence spectrum were: λ_{max} = 496 nm, $\Delta\lambda_{1/2}$ = 64 nm. The shift is 2 nm.

The presence of optical density growth in the absorption spectrum from 400 nm to 350 nm with a calculated transition energy of 2.25 eV, as well as the presence of luminescence of the sample when excited by a laser excitation source at 355 nm shows the presence of deep defect levels. Additional information can be obtained by comparing the luminescence spectra of the synthesized $ZnWO_4$ sample with the reference single-crystal $ZnWO_4$ sample.

The luminescence spectra of a reference sample of single crystal $ZnWO_4$ were measured. The spectrum of the reference sample is shown in Figure 9 (*a*, *b*).



a) b)

Figure 9. Luminescence spectrum of single crystal $ZnWO_4$ sample under luminescence excitation by mercury lamp (*a*) and laser excitation source (*b*)

In the case of the reference $ZnWO_4$ sample, the shift of the luminescence intensity maximum is more than 100 nm between the spectrum obtained under excitation by a mercury lamp λ_{max1} = 490 nm (250–320 nm) and the spectrum under excitation at 355 nm λ_{max2} = 600 nm. This is an additional indirect factor indicating high defectivity of $ZnWO_4$ samples synthesized by the radiation method synthesis. Significant differences in the luminescence spectra of the synthesized sample and the reference sample indicate possible different defects present in the compared samples.

Conclusion

Thus, the surface morphology, EDX analysis, X-ray diffraction spectra and optical properties of $ZnWO_4$ ceramics obtained by radiation synthesis were studied.

On the surface of synthesized ceramic samples there are microparticles of different shapes and sizes. These particles may be the particles from which the synthesis of ceramics was carried out. The distribution of tungsten and oxygen is uniform on the surface of the sample. Zinc is not uniformly distributed over the surface. It is possible that the areas with reduced zinc content may contain various tungsten oxide compounds.

X-ray diffraction measurements confirmed the formation of zinc tungstate $ZnWO_4$ by radiation synthesis.

The optical properties of ceramics were investigated. From the absorption spectra the energy value of transitions related to absorption in the region from 350 to 450 nm. It amounted to 2.25 eV. This energy value may indicate a significant presence of defects in the synthesized sample.

The luminescence spectra of the synthesized sample correspond to those of crystalline $ZnWO_4$. Comparison of luminescence spectra of the synthesized ceramic sample and single-crystal reference sample measured under different conditions of optical excitation indicate significant differences in the luminescence spectra of the synthesized sample and reference sample indicating possible different defects present in the compared samples.

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Г.К. Алпысова, Д.А. Афанасьев, Ж.К. Бакиева, Л.А. Лисицына,
М.Г. Голковский, А.К. Тусупбекова, А.А. Кисабекова, С.Д. Тулеуов

Радиациялық синтез арқылы алынған $ZnWO_4$ керамикасының оптикалық қасиеттері

MWO_4 құрамындағы вольфрамат металдарынан керамика алудың перспективалы әдістерінің бірі — бұл электрондардың қуатты ағындарын пайдалана отырып, радиациялық синтез әдісі. Сәулеленудің бірегей қасиеттерінің арқасында синтез процесін айтарлықтай жеделдету мүмкін болды. $ZnWO_4$ керамикасы энергиясы 1,4-тен 2,5 МэВ-ға дейінгі және қуаты 10–25 кВт/см² жоғары тығыздықтағы электрондар ағынына әсер ету жолымен синтезделуі мүмкін екендігі көрсетілді. Осыған байланысты осы әдіспен алынған $ZnWO_4$ мырыш вольфраматының керамикалық үлгілерінің оптикалық қасиеттеріне зерттеулер жүргізілді. Жұмыста беткі қабаттың морфологиясы, рентген дифракциясының спектрлері және радиациялық синтез әдісімен алынған $ZnWO_4$ керамиканың оптикалық қасиеттері зерттелді. Рентген дифракциясын өлшеу және EDX талдау радиациялық синтез нәтижесінде $ZnWO_4$ мырыш вольфраматы керамикасының қалыптасуын растады. Синтезделген үлгілердің сіңіру, люминесценция, люминесценцияны қоздыру спектрлері өлшенді. Сондай-ақ эталондық монокристалдық үлгідегі люминесценция спектрлері өлшенді. Рентген дифракциясы радиациялық синтез нәтижесінде мырыш ($ZnWO_4$) вольфраматының пайда болуын растады. Синтезделген үлгінің люминесценттік спектрлері кристалды $ZnWO_4$ спектрлерімен сәйкес келеді. Оптикалық қоздырудың әртүрлі жағдайларында өлшенген синтезделген керамикалық үлгінің және монокристалдық эталондық үлгінің люминесценция спектрлерін салыстыру синтезделген үлгінің және эталондық үлгінің люминесценция спектрлеріндегі елеулі айырмашылықтарды және салыстырылатын үлгілерде болуы мүмкін әртүрлі ақауларды көрсетеді.

Кілт сөздер: керамика, люминесценция, радиациялық синтез, мырыш вольфраматы, рентген дифракциясының спектрлері, EDX-талдау, қуат тығыздығы, керамиканың оптикалық қасиеттері.

Г.К. Алпысова, Д.А. Афанасьев, Ж.К. Бакиева, Л.А. Лисицына,
М.Г. Голковский, А.К. Тусупбекова, А.А. Кисабекова, С.Д. Тулеуов

Оптические свойства $ZnWO_4$ керамики, полученной методом радиационного синтеза

Один из перспективных методов получения керамики из металлов вольфрамата состава MWO_4 — это радиационный синтез с использованием мощных потоков электронов. Благодаря уникальным свойствам излучения удалось значительно ускорить процесс синтеза. Было продемонстрировано, что керамика $ZnWO_4$ может быть синтезирована путем воздействия на поток электронов с энергией от 1,4 до 2,5 МэВ и высокой плотностью мощности 10–25 кВт/см². В связи с этим были проведены исследования оптических свойств керамических образцов вольфрамата цинка $ZnWO_4$, полученных данным методом. В статье были изучены морфология поверхности, спектры рентгеновской дифракции и оптические свойства керамики $ZnWO_4$ методом радиационного синтеза. Измерения рентгеновской дифракции и EDX анализ подтвердили формирование керамики вольфрамата цинка $ZnWO_4$ в результате радиационного синтеза. Измерены спектры поглощения, люминесценции, возбуждения лю-

минесценции синтезированных образцов. Также измерены спектры люминесценции эталонного монокристаллического образца. Рентгеновская дифракция подтвердила образование вольфрамата цинка ($ZnWO_4$) в результате радиационного синтеза. Люминесцентные спектры синтезированного образца совпадают со спектрами кристаллического $ZnWO_4$. Сравнение спектров люминесценции синтезированного керамического образца и монокристаллического эталонного образца, измеренные при разных условиях оптического возбуждения, показывает значительные различия в спектрах люминесценции синтезированного образца и эталонного образца и указывает на возможные различные дефекты, имеющиеся в сравниваемых образцах.

Ключевые слова: керамика, люминесценция, радиационный синтез, вольфрамат цинка, спектры рентгеновской дифракции, EDX-анализ, плотность мощности, оптические свойства керамики.

Information about the authors:

Gulnur Alpyssova — PhD, Karaganda Buketov University, University str., 28, Karaganda, Kazakhstan; 100028; e-mail: gulnur-0909@mail.ru; ORCID ID: 0000-0002-7164-2188

Dmitriy Afanasyev — PhD, Karaganda Buketov University, University str., 28, Karaganda, Kazakhstan; 100028; e-mail: a_d_afanasyev@mail.ru; ORCID ID: 0000-0002-0437-5315

Zhanara Bakiyeva — 1st year PhD student, Karaganda Buketov University, University str., 28, Karaganda, Kazakhstan; 100028; e-mail: shanar83@mail.ru ORCID ID: 0009-0005-1949-4535

Liudmila Lisitsyna — PhD, Tomsk State University of Architecture and Building, Solyanaya Sq. 2, Tomsk, Russia.

Mikhail Golkovski — PhD, Senior Researcher, Laboratory of Industrial Electron Accelerators, Budker Institute of Nuclear Physics of Siberian Branch of the Russian Academy of Sciences, Academician Lavrentieva ave., 11, Novosibirsk, Russia, 630090; e-mail: golkovski@mail.ru; ORCID ID: 0000-0003-4399-444X

Ainur Tussupbekova — PhD, Karaganda Buketov University, University str., 28, Karaganda, Kazakhstan; 100028; e-mail: aintus_070482@mail.ru; ORCID ID: 0000-0001-5299-9977.

Assemgul Kissabekova — PhD, Associate Professor, NJSC «Pavlodar Pedagogical University named after A. Margulan», st. Toraigyrova, 58, Pavlodar, Kazakhstan; 140008; e-mail: akisabekova@mail.ru; ORCID ID: 0000-0001-6300-6758

Serik Tuleuov — Student, Karaganda Buketov University, University str., 28, Karaganda, Kazakhstan; e-mail: serik.tu123@gmail.com; ORCID ID: 0009-0002-6009-1672