

Comparative cathodoluminescence of nanostructured AlN and AlN nanotube/CsI scintillator composites

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Abstract. The cathodoluminescence (CL) response of commercially available aluminium nitride (AlN) powder, plasma-synthesized AlN nanoparticles, AlN nanotubes, and AlN nanotube/cesium iodide (CsI) composites was comparatively analysed in the 80–300 K temperature range. The nanostructured material was produced by a highly nonequilibrium direct-current arc-plasma route and contained two characteristic nanotube populations: hollow tubes with diameters of approximately 25–30 nm and thinner, frequently helical tubes with diameters of 2–3 nm; both populations had lengths on the order of 700 nm. At 80 K the commercial AlN powder exhibited a dominant band at 3.47 eV, whereas nanoparticles emitted predominantly near 3.66 eV. The nanotube spectrum was substantially more complex and included components near 2.2, 3.0, and 3.5 eV, indicating a broader distribution of defect- and surface-related radiative channels. A single-channel Mott–Seitz treatment of the temperature-dependent 3.47 eV emission yielded an apparent activation energy of about 24 meV. The absence of a measurable peak shift or linewidth broadening, together with the small fitted energy, indicates that the intensity loss cannot be described as conventional thermal quenching alone and is more consistently explained by carrier migration to non-radiative surface states. Addition of 0.25–0.50 wt.% AlN nanotubes to CsI modified the host emission profile, particularly at 80 K, providing spectral evidence consistent with radiative reabsorption and/or interfacial energy redistribution. The results demonstrate that AlN morphology strongly controls the balance between intrinsic, impurity-related, and surface-mediated recombination and underline the potential of AlN/CsI composites for tunable ultraviolet–visible scintillation materials.

Keywords: aluminium nitride; nanotubes; nanoparticles; cathodoluminescence; surface states; cesium iodide; scintillator composites.

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Introduction

Aluminium nitride (AlN) is a technologically important III–V compound combining a wide direct band gap of approximately 6.2 eV with high thermal conductivity, low thermal expansion, chemical stability, piezoelectricity, and transparency in the visible spectral range. These properties have established AlN as a platform material for ultraviolet optoelectronics, high-power electronics, acoustic-wave devices, thermally conductive electrical insulation, and radiation-resistant components [1–5]. In addition to its stable hexagonal wurtzite structure, AlN can occur in metastable structural forms under highly nonequilibrium synthesis conditions. The electronic and vibrational properties of such nanostructured phases may differ considerably from those of bulk crystals because of reduced dimensions, high surface-to-volume ratio, curvature, local strain, and the enhanced contribution of surfaces and interfaces.

The optical response of AlN is extremely sensitive to non-stoichiometry and residual impurities. Emission well below the fundamental absorption edge is therefore normally dominated by point defects, impurity–defect complexes, and surface states rather than by direct band-to-band recombination. Oxygen is one of the most common residual impurities in AlN and has frequently been associated with blue and near-ultraviolet luminescence. Nevertheless, a particular spectral band should not be assigned to a single microscopic defect without complementary structural, chemical, or first-principles evidence because aluminium vacancies, nitrogen vacancies, oxygen substituting for nitrogen, and their complexes may produce overlapping transitions. In nanostructures this ambiguity is amplified by surface reconstruction and the large density of under-coordinated atoms.

Arc-plasma synthesis provides access to AlN nanoparticles and nanotubes under strongly nonequilibrium conditions. Earlier structural investigations of the material considered here revealed a rare metastable cubic contribution, size-dependent changes in infrared-active vibrations, and a vibrational density of states markedly different from bulk AlN [1–5]. Scanning tunnelling and transmission electron microscopy further showed two characteristic nanotube morphologies: large hollow tubes and much thinner coiled or helical tubes [2]. These structural distinctions are expected to alter carrier localization and the competition between radiative and non-radiative relaxation.

A second motivation for studying AlN nanotubes is their possible use as an active nanoscale component in scintillator composites. Cesium iodide (CsI) is a widely used ionic scintillator host with intense ultraviolet emission and a large stopping power for ionizing radiation [6–8]. Incorporation of AlN nanotubes may introduce additional absorption and emission channels, modify light scattering and reabsorption, and create interfacial states that redistribute excitation energy. However, the interpretation of composite luminescence requires a direct comparison with both pristine CsI and isolated AlN nanostructures under the same excitation conditions.

The present work aimed at a systematic analysis of the morphology-dependent cathodoluminescence (CL) of commercial AlN powder, arc-plasma-synthesized AlN nanoparticles, and AlN nanotubes. Particular attention is paid to the temperature dependence of the 3.47 eV band and to the spectral evolution of CsI containing 0.25 and 0.50 wt.% AlN nanotubes. The purpose is not only to catalogue emission maxima but also to identify which conclusions are directly supported by the spectra and which require additional time-resolved or excitation-selective measurements.

Materials and Methods

AlN nanotubes and nanoparticles were synthesized by a high-temperature, highly nonequilibrium direct-current arc-plasma process described in detail in Refs. [1, 2]. The plasma route produced a broad size distribution typical of rapid nucleation and growth from a high-temperature vapour. Electron-microscopy observations showed larger hollow nanotubes with diameters of approximately 25–30 nm and lengths near 700 nm, together with smaller 2–3 nm tubes of comparable length. The thinner tubes frequently exhibited a helical morphology, whereas the larger objects had a conventional hollow tubular structure. A commercial AlN powder (Aldrich, catalogue No. 24,190-3), containing more than 98% approximately spherical particles with diameters below 10 μm , was used as a bulk-like particulate reference.

For composite measurements, AlN nanotubes were dispersed in CsI at nominal mass fractions of 0.25 and 0.50 wt.%. Pure CsI measured under the same conditions served as the host reference. In the original spectral dataset, the nanotube-containing composite is labelled NT03/CsI; NT03 is retained as the original sample identifier rather than used as a scientific abbreviation.

Cathodoluminescence spectra were recorded under electron-beam excitation with an electron energy of 9 keV and an emission current of 150 μ A over the temperature interval 80–300 K. Emitted light was dispersed by a double-prism monochromator and detected with an FEU-106 photomultiplier covering approximately 200–800 nm. The spectral energy resolution was better than 30 meV. Instrumental response was corrected using a calibrated light source. Because the displayed comparative AlN spectra are normalized, conclusions concerning absolute photon yield cannot be drawn from those curves.

The thermal behaviour of the dominant 3.47 eV band in commercial AlN was evaluated using the conventional one-channel Mott-Seitz expression:

$$I(T) = I_0 / \{1 + C \exp[-E_a/(kBT)]\},$$

where $I(T)$ is the integrated luminescence intensity at temperature T , I_0 is the low-temperature intensity, C is a pre-exponential factor, E_a is the apparent activation energy, and kB is the Boltzmann constant. This phenomenological model assumes competition between a radiative channel and a thermally activated non-radiative channel. Its physical validity must therefore be tested against simultaneous changes in peak position, spectral width, and line shape.

Results and Discussion

1. Morphology-dependent emission of AlN

Figure 1 compares the normalized 80 K CL spectra of the commercial AlN powder, nanoparticles, and nanotubes. The commercial AlN powder is dominated by a relatively well-defined band centred at approximately 3.47 eV (about 357 nm). This band lies far below the 6.2 eV band gap and is therefore defect-mediated. Oxygen-related defects are a plausible contributor because oxygen is a ubiquitous impurity in AlN and oxygen-associated blue emission has been reported extensively [6]. At the same time, the spectrum by itself does not discriminate between isolated oxygen substitutions and complexes involving aluminium vacancies or other native defects.

The nanoparticle spectrum is shifted to higher photon energy and is centred near 3.66 eV (approximately 339 nm). A simple quantum-confinement explanation would be inadequate because the reported particles and nanotubes are generally much larger than the characteristic electronic length scales of AlN. More credible contributions include differences in phase content, local strain, defect charge state, stoichiometry, and surface band bending. The higher-energy maximum may reflect a changed distribution of donor–acceptor pair separations or a different balance between bulk-like and surface-localized recombination. The independent observation of a metastable cubic phase and shifted vibrational modes supports this interpretation, although phase-resolved luminescence would be required for a definitive assignment.

The nanotube spectrum has the broadest and most structured profile, with at least three components near 2.2, 3.0, and 3.5 eV. The low-energy 2.2 eV band (approximately 564 nm) is characteristic of a deep recombination channel and is likely associated with strongly localized defect states or surface-related complexes. The intermediate 3.0 eV component (approximately 413 nm) may originate from another family of defect complexes or from interface-like states generated by curvature and reconstructed surfaces. The component near 3.5 eV overlaps the spectral region of the commercial AlN powder band and may contain oxygen-related or native-defect contributions. The coexistence of hollow and helical tubes, with diameters differing by an order of magnitude, naturally produces a distribution of local environments and can account for the complex line shape.

Table 1 – Comparative summary of the investigated AlN-containing samples and their principal CL features.

Sample	Structural/ morphological context	Main CL features	Most conservative interpretation
Commercial AlN powder	>98% spherical particles; diameter <10 μ m	Dominant band at 3.47 eV	Defect-mediated emission; oxygen-associated defects are plausible but not uniquely proven

Sample	Structural/ morphological context	Main CL features	Most conservative interpretation
AlN nanoparticles	Arc-plasma product; nonequilibrium phase/ defect structure	Broad maximum near 3.66 eV	Modified defect population, strain, phase content, and surface electronic structure
AlN nanotubes	25–30 nm hollow tubes and 2–3 nm helical tubes; length ~700 nm	Components near 2.2, 3.0, and 3.5 eV	Multiple deep, surface-related, and impurity/native-defect recombination channels
CsI + 0.25 wt.% AlN nanotubes	Dilute nanotube/CsI composite	Host profile modified; additional low-energy contribution is visible, especially at 295 K	Spectral redistribution consistent with reabsorption and/or interfacial energy transfer
CsI + 0.50 wt.% AlN nanotubes	Higher nanotube loading	Broad structured emission at 80 K	Stronger contribution of nanotube/ interface states; quantitative yield not available

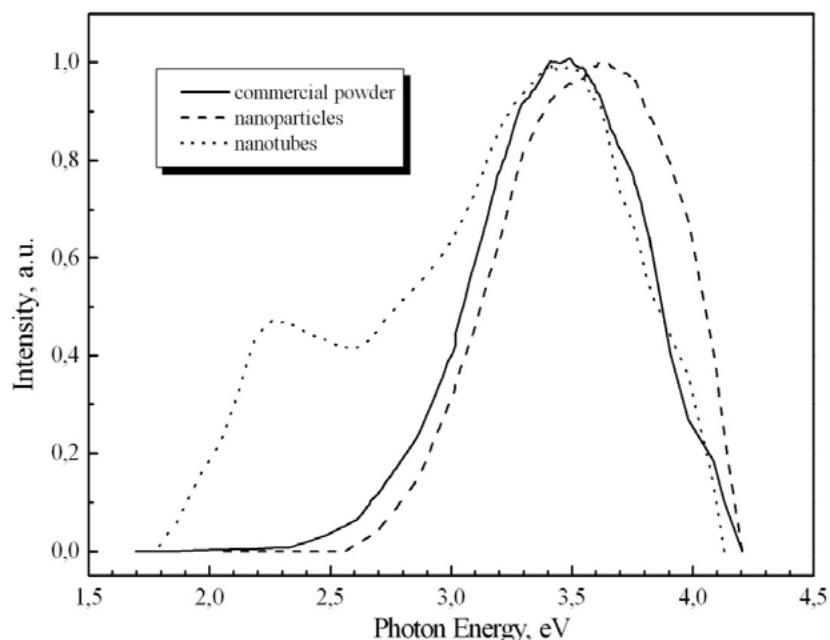


Figure 1. Normalized cathodoluminescence spectra of commercial AlN powder, AlN nanoparticles, and AlN nanotubes recorded at 80 K. The commercial powder is dominated by a band near 3.47 eV, nanoparticles emit mainly near 3.66 eV, and nanotubes show a multicomponent spectrum with bands near 2.2, 3.0, and 3.5 eV.

2. Temperature dependence of CL

The integrated intensity and half-width of the commercial AlN powder band were analysed between 80 and 300 K. No significant temperature-induced broadening or systematic spectral shift of the 3.47 eV maximum was observed within the resolution of the experiment. Fitting the intensity decrease with the one-channel Mott–Seitz equation produced an apparent activation energy of approximately 24 meV. This energy is close to the thermal energy available near room temperature and is too small to support a unique assignment to escape from a well-defined deep defect level over the full 80–300 K interval.

More importantly, the assumptions of the simple Mott–Seitz model are not fully satisfied. Conventional thermal depopulation of a localized emitting state often changes the relative populations of nearby states and may be accompanied by a measurable evolution of the peak position, linewidth, or spectral shape. Their absence suggests

that the observed intensity decrease is governed by a spatial transport process rather than by thermal emptying of one isolated luminescence centre. Electron-hole pairs generated in the penetration volume of the electron beam can migrate before recombination. As temperature increases, diffusion and trapping kinetics may favour migration toward particle surfaces, where dangling bonds, adsorbates, and reconstruction-related states provide efficient non-radiative pathways. The extracted 24 meV should therefore be regarded as an effective transport or trapping scale, not as a definitive microscopic defect ionization energy.

The stronger room-temperature decrease observed for the nanostructured samples is consistent with this picture. Their surface-to-volume ratio is much larger than that of the commercial micropowder, and their curved surfaces can contain a high density of structurally inequivalent sites. Electron-phonon interactions and self-absorption may further reduce the detected emission. Separating these mechanisms would require absolute quantum-yield measurements, temperature-resolved decay kinetics, and a controlled comparison of powders with known surface area and oxygen content.

3. Luminescence of AlN nanotube/CsI composites

Figure 2 compares pure CsI with the composite containing 0.25 wt.% AlN nanotubes at 295 and 80 K. Pure CsI shows a broad ultraviolet emission profile whose relative intensity and shape change on cooling. Introduction of a small amount of AlN nanotubes modifies the spectrum rather than merely scaling the host intensity. At 295 K the composite retains a host-related maximum in the ultraviolet but develops a more pronounced low-energy wing. At 80 K the structured ultraviolet emission becomes stronger and the difference between the host and composite remains observable. Since the AlN nanotubes themselves emit across approximately 2.2–3.5 eV, overlap exists between the CsI emission and absorption or defect-state excitation channels of the nanotubes.

The spectral modification is consistent with two non-exclusive mechanisms. First, photons emitted by CsI may be absorbed by AlN nanotubes and re-emitted through nanotube defect states, representing radiative energy transfer or luminescence reabsorption. Second, carriers or excitations generated close to a CsI/AlN interface may relax through interface-mediated states before recombination. The available steady-state spectra demonstrate redistribution of the emitted intensity but do not independently establish the transfer pathway. A rigorous distinction would require excitation spectra monitored at the nanotube bands, time-resolved measurements showing correlated decay components, and comparison of absolute light yields after correction for scattering and optical density.

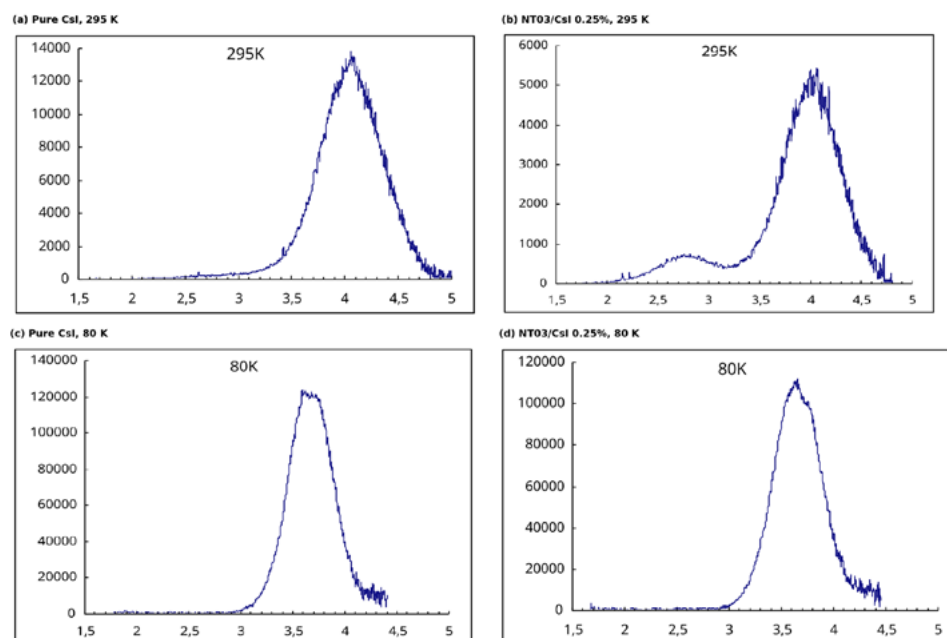


Figure 2. Cathodoluminescence spectra of pure CsI and CsI containing 0.25 wt.% AlN nanotubes at 295 K and 80 K. The panels reproduce the original spectral data on common axes for direct comparison. The composite exhibits a modified ultraviolet profile and an enhanced low-energy contribution.

The 0.50 wt.% composite measured at 80 K (Figure 3) displays a broad, structured emission extending from the near-ultraviolet toward the visible range. Compared with the lower-loading sample, the larger nanotube fraction increases the probability that host emission or migrating excitations interact with an AlN surface. At the same time, an excessive nanotube concentration may increase optical scattering, self-absorption, and the density of non-radiative interfaces. The present dataset does not include an absolute intensity calibration between all compositions, so an optimum nanotube loading cannot yet be identified. The spectra should therefore be interpreted as evidence of tunability rather than proof that 0.50 wt.% maximizes scintillation efficiency.

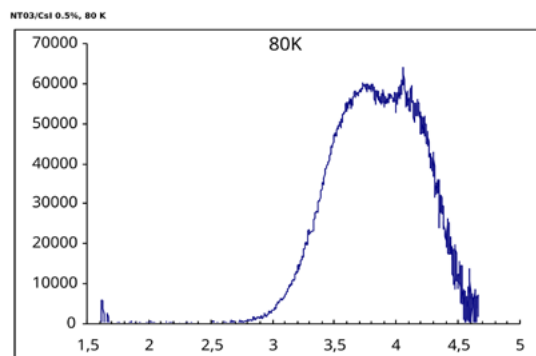


Figure 3. Cathodoluminescence spectrum of CsI containing 0.50 wt.% AlN nanotubes at 80 K. The broad structured profile indicates simultaneous contributions from the CsI host, nanotube defect states, and/or interfacial relaxation channels. Source: supplied poster dataset.

4. Implications for defect physics and scintillator design

The combined data show that morphology is not a secondary variable in AlN luminescence. Commercial microparticles, nanoparticles, and nanotubes exhibit different emission maxima even under identical electron-beam excitation and temperature. The observation is consistent with a redistribution of defects and charge states caused by nonequilibrium synthesis and by the large contribution of surfaces. The coexistence of multiple nanotube diameters and morphologies additionally broadens the ensemble response. Consequently, future optimization should treat phase composition, oxygen concentration, surface passivation, and tube diameter distribution as independent synthesis parameters.

For scintillator applications, a broad nanotube emission can be advantageous when spectral matching to a photodetector is required, but it can also indicate a high concentration of deep traps that slow energy transport or promote non-radiative loss. The most promising composite would therefore not necessarily be the one with the strongest visible shoulder; it would be the composition that maximizes the number of emitted photons per unit deposited energy while retaining an acceptable decay time and radiation stability. Measurements of radioluminescence under X-ray or gamma excitation, scintillation decay kinetics, thermally stimulated luminescence, and dose-dependent stability are needed to establish this balance.

Conclusions

A comparative analysis of commercial AlN powder, arc-plasma-synthesized nanoparticles, and AlN nanotubes demonstrates a pronounced morphology dependence of cathodoluminescence. At 80 K the commercial AlN powder is characterized by a dominant defect-related band at 3.47 eV, while nanoparticles emit mainly near 3.66 eV. AlN nanotubes exhibit a substantially more complex spectrum with components near 2.2, 3.0, and 3.5 eV, reflecting the combined influence of deep defects, impurities, structural nonequilibrium, and surface states. The apparent activation energy of 24 meV obtained from a one-channel Mott-Seitz fit to the commercial AlN powder intensity should not be interpreted as a unique defect ionization energy. The lack of measurable peak shift and linewidth broadening supports a model in which thermally assisted carrier migration to non-radiative surface sites controls much of the intensity loss.

Incorporating 0.25–0.50 wt.% AlN nanotubes into CsI modifies the host CL spectrum and introduces or enhances lower-energy emission. These changes are consistent with radiative reabsorption and/or interfacial energy redistribution between CsI and AlN nanotubes. However, steady-state normalized spectra alone cannot quantify transfer efficiency or establish an optimal loading. Absolute light-yield measurements, excitation-selective spectroscopy, decay kinetics, and controlled structural and chemical characterization are required for further development. Within these limitations, the results identify AlN nanotubes as an effective spectral-modification component and provide a basis for designing ultraviolet–visible scintillator composites with tunable emission.

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Conflicts of Interest

The authors declare no conflict of interest.

Author Contributions

Conceptualization, **Popov A.I.**; methodology, **Savchyn V.P.**, **Balasubramanian C.** and **Konuhova M.**; investigation, **Savchyn V.P.**, **Balasubramanian C.** and **Konuhova M.**; formal analysis, **Savchyn V.P.**, **Popov A.I.** and **Konuhova M.**; visualization, **Savchyn V.P.** and **Konuhova M.**; writing—original draft preparation, **Savchyn V.P.** and **Konuhova M.**; writing—review and editing, all authors; supervision, **Popov A.I.** All authors have read and agreed to the published version of the manuscript.

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Сравнительная катодолуминесценция наноструктурированного AlN и сцинтилляционных композитов AlN-нанотрубки/CsI

Аннотация. Проведён сравнительный анализ катодолуминесценции промышленного порошка нитрида алюминия (AlN), синтезированных дуговым плазменным методом наночастиц и нанотрубок AlN, а также композитов AlN-нанотрубки/иодид цезия (CsI) в диапазоне температур 80–300 К. При 80 К промышленный AlN характеризуется полосой с максимумом 3,47 эВ, наночастицы — излучением около 3,66 эВ, тогда как спектр нанотрубок содержит компоненты около 2,2, 3,0 и 3,5 эВ. Различия связаны с неравновесной фазовой и дефектной структурой, кривизной и высоким вкладом поверхностных состояний. Формальная аппроксимация температурного тушения полосы 3,47 эВ моделью Мотта–Зейтца дала энергию активации около 24 мэВ. Отсутствие заметного температурного сдвига и уширения полосы показывает, что снижение интенсивности нельзя описать только обычным термическим опустошением центра; более вероятно миграция носителей к безызлучательным поверхностным состояниям. Добавление 0,25–0,50 мас.% нанотрубок AlN изменяет профиль люминесценции CsI, что согласуется с переизлучением, реабсорбцией и/или межфазным перераспределением энергии. Для количественного определения эффективности переноса необходимы измерения абсолютного световыхода и кинетики затухания.

Ключевые слова: нитрид алюминия; нанотрубки; наночастицы; катодолуминесценция; поверхностные состояния; иодид цезия; сцинтилляционные композиты.

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Нанокұрылымды AlN және AlN нанотүтікшелері/CsI сцинтилляциялық композиттерінің салыстырмалы катодолуминесценциясы

Аңдатпа. 80–300 К температура аралығында өнеркәсіптік алюминий нитриді (AlN) ұнтағының, доғалық плазмалық әдіспен синтезделген AlN нанобөлшектері мен нанотүтікшелерінің, сондай-ақ AlN нанотүтікшелері/цезий иодиді (CsI) композиттерінің катодолуминесценциясы салыстырмалы түрде зерттелді. 80 К температурада өнеркәсіптік AlN үшін 3,47 эВ максимумды жолақ, нанобөлшектер үшін шамамен 3,66 эВ сәуле шығару байқалды, ал нанотүтікшелер спектрінде 2,2, 3,0 және 3,5 эВ маңындағы бірнеше құрамдас бар. Бұл айырмашылықтар тепе-тең емес фазалық және ақаулық құрылыммен, қисықтықпен және беттік күйлердің жоғары үлесімен байланысты. 3,47 эВ жолағының температуралық сөнуін Мотт–Зейтц моделімен формалды жуықтау шамамен 24 мэВ белсендіру энергиясын берді. Жолақ орны мен енінің айқын өзгермеуі интенсивтіліктің төмендеуін тек кәдімгі термиялық сөнумен түсіндіруге болмайтынын және тасымалдаушылардың сәуле шығармайтын беттік күйлерге миграциясы маңызды екенін көрсетеді. CsI матрицасына 0,25–0,50 масс.% AlN нанотүтікшелерін енгізу сәуле шығару профилін өзгертеді, бұл қайта

жұтылу және/немесе фазаралық энергияның қайта бөлінуімен үйлеседі. Энергия тасымалының тиімділігін сандық бағалау үшін абсолюттік жарық шығымын және өшу кинетикасын өлшеу қажет.

Түйін сөздер: алюминий нитридi; нанотүтікшелер; нанобөлшектер; катодолуминесценция; беттік күйлер; цезий иодиді; сцинтилляциялық композиттер.

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