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1. Introduction

Accurate temperature measurement is important in many applications, and temperature measurement at the micro/nano scale is a particularly challenging task, because measuring the temperature in the field, such as in intracellular or micro/nano electronics is impractical when using a conventional thermometer.^{1–5} To solve this problem, different temperature measurement techniques have been developed, including nanolithographic thermometry, the scanning thermal microscope method, the resistance thermometry technique, optical techniques, and so forth.⁶⁻⁸ Among these different techniques, optical temperature sensors based on the fluorescence intensity ratio (FIR) technique have attracted great attention,^{2,9–21} as this kind of sensor has the advantages of a rapid response time and immunity to a harsh environment when compared to traditional contact thermometers.^{2,10,11} Er³⁺ is commonly used in the above mentioned temperature sensors,¹²⁻¹⁶ because the two thermally coupled ²H_{11/2} and ⁴S_{3/2} levels of Er³⁺ give an intense green emission and their relative emission shows an obvious change depending on

Synthesis, optical properties and application of $Y_7O_6F_9$:Er³⁺ for sensing the chip temperature of a light emitting diode

Deyin Wang, (1)* Pengpeng Zhang, Qiang Ma, Jiachi Zhang (1)* and Yuhua Wang (1)*

Submicron-sized Er^{3+} doped $Y_7O_6F_9$ phosphors were synthesized *via* the precipitation method and a subsequent annealing process. The influence of the Er^{3+} doping concentrations and the heating temperature on the luminescence of $Y_7O_6F_9$: Er^{3+} was investigated under the excitation of the $Er^{3+} \, {}^4G_{11/2}$ level at 378 nm. Upon raising the Er^{3+} doping concentration, both the intensity and lifetime of the Er^{3+} green emission at 547 nm originating from ${}^4S_{3/2} \rightarrow {}^4I_{15/2}$ decrease, which is proved to be caused by energy transfer *via* cross relaxation between two neighboring Er^{3+} ions. The thermal sensing properties of $Y_7O_6F_9$: Er^{3+} were evaluated using the temperature dependent intensity ratio between the ${}^2H_{11/2} - {}^4I_{15/2}$ and ${}^4S_{3/2} - {}^4I_{15/2}$ transitions of Er^{3+} under 378 nm excitation. The experimental results show that the thermal sensitivity decreased with an increase in the Er^{3+} doping content, which is caused by the increased energy transfer probability among Er^{3+} . The increased energy transfer of Er^{3+} reduces the thermalized population of Er^{3+} in the ${}^2H_{11/2}$ levels, which in turn decreases the thermal sensitivity. The applicability of Er^{3+} doped $Y_7O_6F_9$ as a thermal sensor was demonstrated by measuring the chip temperature of a 1 W InGaN type near-ultraviolet light emitting diode (n-UV LED).

the temperature. However, a careful look at the existing literature reveals that the previously reported Er³⁺-based thermal sensor mainly utilized the up-conversion (anti-stokes luminescence) luminescence of Er^{3+} , 12-16 and a few works examined the thermal sensibility by using the Stokes luminescence from Er³⁺. It is well known that the up-conversion luminescence is a non-linear process and generally has a low efficiency (the efficiency is strongly dependent on the power of the incident light).¹⁰ Apart from the low efficiency of the up-conversion, the thermal effect caused by high powered near infrared light excitation (e.g. 980 nm) may make the measured temperature deviate from the actual temperature.^{11,12} On the contrary, the efficiency of the Stokes luminescence is independent of the excitation power and the thermal effect caused by the excitation light in Stokes luminescence can be ignored. Meanwhile, whether or not the sensor utilizes the up-conversion or Stokes luminescence, host materials with a low phonon energy are preferred, as such host materials can depress the multiphonon relaxation process, resulting in a higher luminescence efficiency from the luminescent center, which is beneficial for the accuracy of the measurement. In this work, we synthesized different concentrations of Er³⁺ doped Y₇O₆F₉ via a co-precipitation method, and investigated the effects of the Er³⁺ doping concentrations on the luminescence and thermal sensitivity of Y₇O₆F₉:Er³⁺ under excitation of the Er³⁺ ${}^{4}G_{11/2}$ level at 378 nm. Finally, the potential use of $Y_{7}O_{6}F_{9}:Er^{3+}$ as

National & Local Joint Engineering Laboratory of Light-conversion Materials and Technology, School of Physical Science and Technology, Lanzhou University, Lanzhou 730000, China. E-mail: wangdy@lzu.edu.cn, zhangjch@lzu.edu.cn, wyh@lzu.edu.cn

an efficient optical temperature sensing material has been demonstrated by determining the chip temperature of a 1 W near-ultraviolet light emitting diode (n-UV LED).

2. Experimental

2.1 Materials and synthesis

The raw materials for Y_2O_3 (99.9%), Er_2O_3 (99.9%) and NH_4F (99.99%) were purchased from Aladdin Chemical reagent company and used without further purification.

Powder samples $Y_7O_6F_9:xEr^{3+}$ (x = 0, 1%, 3% and 5%) were prepared using a co-precipitation method. The Er^{3+} doping concentration refers to the nominal concentration in mol%. In detail, stock solutions of 0.5 mmol ml⁻¹ Y(NO₃)₃ and 0.005 mmol ml⁻¹ Er(NO₃)₃ were prepared in advance by dissolving Y_2O_3 and Er_2O_3 in hot nitric acid, and the residual nitric acid was evaporated by heating. The weighed trivalent lanthanide nitrate solution was added dropwise into the NH₄F solution by using a separatory funnel under continuous stirring. The resulting white precipitate was separated by centrifugation and dried at 80 °C in an oven for 12 h. Finally, the precipitate was annealed at 600 °C for 2 h followed by 800 °C for 2 h in air with intermediate grounding.

2.2 Characterization

The X-ray diffraction (XRD) data were measured using a Bruker D2 Phaser powder X-ray diffractometer. The photoluminescence (PL) and photoluminescence excitation (PLE) spectra were measured using a Fluorlog-3 spectrophotometer. The decay curves and quantum efficiency were measured using the FLS 980 spectrometer. Scanning electron microscopy (SEM) was recorded by using the Hitachi S-4800 electron microscope. Transmission electron microscope (TEM), high-resolution transmission electron microscope (HRTEM) and selected area electron diffraction (SAED) images were taken on a JEM-2010 electron microscope. Elemental mapping was obtained on a TECNAI G2 TF20 electron microscope. The Er³⁺ doping concentration in all samples was determined by using inductively coupled plasma-atomic emission spectrometry (ICP-AES) (PlasmaQuant 9000). The electroluminescent (EL) spectrum was measured using an EVERFINE spectrophotometer with an integral sphere under ambient conditions at room temperature.

3. Results and discussion

3.1 Morphology and phase identification

The structure of $Y_7O_6F_9$ consists of a single YO⁺ layer sandwiched between F⁻ layers. There are two coordination environments for Y³⁺ in Y₇O₆F₉, *viz.* one site is coordinated by four O²⁻ and three F⁻ anions, the other site is coordinated by four O²⁻ and four F⁻ anions.²² In view of same valence and similar ionic radius of Er³⁺ (*r* = 89 pm) and Y³⁺ (90 pm),²³ the Er³⁺ ions are expected to occupy the Y sites in Y₇O₆F₉. Fig. 1a shows the XRD patterns of the precursor prepared using the precipitation method and the samples after being annealed at different temperatures for 2 h.



Fig. 1 (a) XRD patterns of the precursor prepared using the precipitation method and the samples annealed at different temperatures. (b) XRD patterns of $Y_7O_6F_9$:xEr³⁺.

The as-fabricated precursor dried at 80 °C is identified as YF₃·1.5NH₃ (JCPDS No. 281449). When the precursor is subject to post-annealing at 600 °C, it transforms into the orthorhombic phase YF_3 (JCPDS No. 701935). With further increasing of the post annealing temperature to 800 °C, the final product becomes an orthorhombic phase $Y_7O_6F_9$ (JCPDS No. 801126), and when the post annealing temperature is increased up to 1000 °C, $Y_7O_6F_9$ is converted into a mixture of YOF (JCPDS No. 712100) and Y_2O_3 (JCPDS No. 895592). Based on the XRD results, the Er^{3+} doped precursor samples were firstly annealed at 600 °C then at 800 °C with intermediate grinding to obtain the pure phase of Y₇O₆F₉. Fig. 1b shows the XRD patterns of Y₇O₆F₉ doped with different concentrations of Er³⁺. The diffraction patterns for all of the Er³⁺ doped samples were found to be identical to the reference data (JCPDS No. 801126) and no other impurities were observed, indicating the introduction of Er³⁺ did not change the crystal structure Y₇O₆F₉. In addition, because the ionic radius of Er^{3+} (*r* = 89 pm) is very close to that of Y^{3+} (*r* = 90 pm)²³ and owing to the successful substitution of Y3+ by Er3+, the diffraction pattern demonstrated almost has no changes upon doping with Er^{3+} . To give the quantitative data for the doping amount of Er^{3+}

Table 1 Nominal and measured ${\rm Er}^{3+}$ doping concentrations in different samples

Sample	Nominal concentration of Er ³⁺ (wt%)	Measured concentration of Er^{3+} (wt%)
$Y_7O_6F_9:0.5 \text{ mol}\%\text{Er}^{3+}$	0.66	0.63
Y ₇ O ₆ F ₉ :1 mol%Er ³⁺	1.30	1.27
Y ₇ O ₆ F ₉ :3 mol%Er ³⁺	3.88	3.75
Y ₇ O ₆ F ₉ :5 mol%Er ³⁺	6.38	6.51



Fig. 2 SEM (a), TEM and HRTEM images (b and c), and SAED pattern (d) for $Y_7O_6F_9{:}1\% Er^{3+}.$

in each sample, spectral analysis using ICP-AES technique was conducted. The obtained results are listed in Table 1, from which it can be seen that the measured Er^{3+} concentration in each sample was close to the corresponding nominal concentration.

Fig. 2a-d shows the SEM, TEM and HRTEM images together with the SAED pattern of $Y_7O_6F_9$:1%Er³⁺, respectively. It can be seen from Fig. 2a and b that the obtained sample has an irregular shape with the presence of agglomerates, which are probably caused by the subsequent high temperature calcinations. From some of separated particles presented in Fig. 2a and b, it can be seen that the agglomerates are composed of particles with a size of around 500-700 nm. The HRTEM image (Fig. 2c) clearly exhibits lattice fringes with an interval of 0.315 nm, corresponding to the (171) lattice plane of the orthorhombic phase $Y_7O_6F_9$. The well-resolved lattice fringes indicate the high crystallinity of the obtained sample. The bright diffraction spots in the SAED pattern (Fig. 2d) further reveal the high crystallinity of $Y_7O_6F_9$: Er^{3+} , and this is probably because the area of the sample selected by the aperture in the TEM contains only a few crystals, the SAED pattern is made up of spots. The elemental mapping of $Y_7O_6F_9$:1%Er³⁺ is shown Fig. 3, which clearly indicates the uniform distribution of the four elements (F, O, Y, and Er) over the whole particle.

3.2 Concentration dependent luminescence of Y₇O₆F₉:Er³⁺

Fig. 4 shows the PLE and PL spectra of $Y_7O_6F_9$ doped with different amounts of Er^{3+} . All of the PLE spectra are same except for the intensity. The sharp absorption peaks at 365, 378, 450, 490 and 520 nm observed in the PLE spectra are ascribed



Fig. 3 Elemental mapping of $Y_7O_6F_9$:1%Er³⁺.



Fig. 4 (a) PLE and (b) PL spectra for $Y_7O_6F_9:xEr^{3+}$

to the transitions of Er^{3+} from its ground state $^{4}\mathrm{I}_{15/2}$ to its excited states $^{4}\mathrm{G}_{9/2}/^{2}\mathrm{K}_{15/2},~^{4}\mathrm{G}_{11/2},~^{4}\mathrm{F}_{5/2},~^{4}\mathrm{F}_{7/2}$ and $^{2}\mathrm{H}_{11/2}$, respectively. 18,24,25 Upon the strongest absorption at 378 nm, corresponding to the Er^{3+} $^{4}\mathrm{I}_{15/2} \rightarrow {}^{4}\mathrm{G}_{11/2}$ transition, all of the emission spectra for the samples are composed of an intense green emission at 547 nm and a weaker

red emission at 670 nm, originating from the $^2H_{11/2}/^4S_{3/2} \rightarrow {}^4I_{15/2}$ and ${}^4F_{9/2} \rightarrow {}^4I_{15/2}$ transitions of Er^{3+} , respectively. 21,22 The overall emission intensity increases with an increased Er^{3+} doping concentration because of the increased number of luminescent centers. When the Er^{3+} doping concentration exceeds 1%, its emission intensity starts to decrease owing to the concentration quenching effect caused by the energy transfer. The internal quantum efficiency of the 1%Er^{3+} doped sample was determined to be 40.1%.

To provide further evidence for the occurrence energy transfer in $Y_7O_6F_9$:xEr³⁺, the luminescence decay curves monitoring the green emission at 547 nm under 378 nm excitation were measured and the results are shown in Fig. 5. The decay curves were normalized on the maximum intensity and plotted as a semi-logarithmic plot. The decay curves were fitted using a double exponential function:²⁶

$$I(t) = A_1 \exp(-t/\tau_1) + A_2 \exp(-t/\tau_2)$$
(1)

In which *I* is the luminescence intensity and A_1 and A_2 are the fractional contributions of the decay lifetimes τ_1 and τ_2 . The average lifetime τ is determined by:

$$\tau = \frac{A_1 \times \tau_1^2 + A_2 \times \tau_2^2}{A_1 \times \tau_1 + A_2 \times \tau_2}$$
(2)

The fitting parameters of each decay curve are summarized in Table 2. The two decay components correspond to Er^{3^+} in two different sites in Y₇O₆F₉. When the Er^{3^+} doping concentrations are increased, the lifetimes of both of the Er^{3^+} emissions from the



Fig. 5 Decay curves (dotted lines) for the ${\rm Er}^{3+}$ emission at 547 nm in $Y_7O_6F_9:x{\rm Er}^{3+}$ under excitation at 378 nm. The solid lines are the fitted results.

Table 2 Fitting parameters for the decay curve for $Y_7O_6F_9{:}xEr^{3+}$ under excitation at 378 nm

x (%)	A_1	$\tau_1 \text{ (ms)}$	A_2	$\tau_2 \text{ (ms)}$	$T(\mathrm{ms})$	R^2
0.5	0.393	0.058	0.607	0.196	0.173	0.9997
1	0.511	0.045	0.488	0.177	0.149	0.9996
3	0.842	0.025	0.158	0.090	0.051	0.9996
5	0.925	0.017	0.075	0.067	0.029	0.9995

two sites decrease (the average lifetime of the Er^{3+} emission at 547 nm decreases from 173 µs at x = 0.5% to 29 µs at x = 5%), which is caused by the increased energy transfer among the Er^{3+} - Er^{3+} and Er^{3+} -defects.

To further clarify the energy transfer process in the Er³⁺doped $Y_7O_6F_9$, the Vis-NIR-IR emission spectra for $Y_7O_6F_9$: xEr^{3+} excited at 378 nm were measured (Fig. 6a). The near-infrared emission at around 1000 nm and the infrared emission at around 1550 nm were assigned to transitions from the excited states ${}^{4}I_{11/2}$ and ${}^{4}I_{13/2}$ ₂ to the ground state ⁴I_{15/2} of Er³⁺ ions, respectively.^{24,25} When the Er³⁺ doping contents are increased, the visible green emission at 547 nm deceases while the infrared emissions at 1000 nm and 1550 nm increase. This phenomena can be rationalized by a cross relaxation energy transfer between the two neighboring Er³⁺ ions $via (Er^{3+4}S_{3/2} \rightarrow {}^{4}I_{13/2}:Er^{3+4}I_{15/2} \rightarrow {}^{4}I_{11/2})$ as indicated by the pink dotted line shown in Fig. 6b. Owing to the energy migration process, the population of the Er³⁺ in ⁴S_{3/2} state decreases, while that in ${}^{4}I_{13/2}$ and ${}^{4}I_{11/2}$ increases, as a consequence, the green emission from the ⁴S_{3/2} state decreases while the two infrared emissions increase accordingly.24,25 These results suggest that the cross relaxation process between the two nearby Er³⁺ is the main depopulation process of the Er^{3+} at ${}^{4}S_{3/2}$ state.



Fig. 6 (a) Vis-NIR-IR PL spectra for $Y_7O_6F_9$:xEr³⁺ under excitation at 378 nm. (b) Energy level diagram for Er³⁺ and the proposed luminescence process under excitation in the Er³⁺ ${}^4G_{11/2}$ level.

3.3 Thermal sensing performance of Y₇O₆F₉:Er³⁺

To examine the application potential of Er^{3^+} doped $Y_7O_6F_9$ for determining temperature, the dependence of the Er^{3^+} green emission originating from the ${}^{2}\mathrm{H}_{11/2}{}^{4}\mathrm{S}_{3/2} \rightarrow {}^{4}\mathrm{I}_{15/2}$ transitions on temperature in a temperature region from 300 to 550 K was measured. Temperature sensing using Er^{3^+} is based on the measurement of the intensity ratio between the luminescence from two closely separated energy levels, ${}^{2}\mathrm{H}_{11/2}$ and ${}^{4}\mathrm{S}_{3/2}$, whose relative population is determined by the Boltzmann distribution:¹⁶⁻¹⁹

$$FIR = I_{522nm}/I_{547nm} = C \exp(-\Delta E/kT)$$
(3)

In which I_{522nm} and I_{547nm} are the integrated intensities of the ${}^{2}H_{11/2} \rightarrow {}^{4}I_{15/2}$ and ${}^{4}S_{3/2} \rightarrow {}^{4}I_{15/2}$ transitions of Er^{3+} , respectively, *C* is a constant, ΔE is the energy difference between ${}^{2}H_{11/2}$ and ${}^{4}S_{3/2}$, and *k* is the Boltzmann constant.¹⁶⁻¹⁹ Fig. 7a illustrates the green emission spectra obtained upon raising the temperature for the lowest Er^{3+} concentration, the 0.5% doped sample, which was normalized to the strongest intensity of the ${}^{4}S_{3/2} \rightarrow {}^{4}I_{15/2}$ emission peak to better show the intensity variation from the ${}^{2}H_{11/2}$ and ${}^{4}S_{3/2}$ levels. It can be seen from Fig. 7a that the intensity ratio between the two levels gradually increases with the



Fig. 7 (a) Temperature dependent green emission spectra of $Y_7O_6F_9$:0.5% Er^{3+} . (b) Dependence of FIR on temperature in $Y_7O_6F_9$:X Er^{3+} . The intensity of the $Er^{3+2}H_{11/2} \rightarrow {}^4l_{15/2}$ transition was integrated from 510 nm to 540 nm, while that of the ${}^4S_{3/2} \rightarrow {}^4l_{15/2}$ transition was integrated from 540 nm to 580 nm.



Fig. 8 Thermal sensitivity of $Y_7O_6F_9:xEr^{3+}$ in the temperature range from 300 to 550 K. The points show the experimental values and the solid lines show the theoretical values.

increasing temperature, which can be explained using eqn (3). Fig. 7b shows the dependence of the FIR between the ${}^{2}H_{11/2} \rightarrow$ ${}^{4}I_{15/2}$ and ${}^{4}S_{3/2} \rightarrow {}^{4}I_{15/2}$ emission bands of Er^{3+} on the absolute temperature for the different concentrations of Er^{3+} doped $Y_7O_6F_9$. It can be seen from Fig. 7b that the FIR increases slowly in the doped sample with a higher concentration of Er³⁺, which is considered to be caused by the re-absorption of Er³⁺. There is an obvious spectral overlap between the emission $^2H_{11/2}$ \rightarrow $^4I_{15/2}$ and absorption ${}^{4}I_{15/2} \rightarrow {}^{2}H_{11/2}$ of Er³⁺ in the 500–530 nm region (see Fig. 4a and b). When the Er³⁺ doping concentrations and heating temperature increase, the distance between the Er³⁺ ions is reduced, and as a consequence of thermal assisted energy transfer, re-absorption among the neighboring Er^{3+} is increased. Therefore, at a higher given temperature, the FIR is decreased with the increasing Er^{3+} doping concentration. In addition, the dependence of the FIR on temperature for the different Er³⁺ doped samples can be well fitted by using eqn (3), and the corresponding fitted function is listed in Fig. 7b. In all samples, the ΔE determined from the fitting results is close ($\Delta E_{0.5\%}$ = 730 cm⁻¹, $\Delta E_{1\%}$ = 726 cm⁻¹, $\Delta E_{3\%} = 690 \text{ cm}^{-1}$ and $\Delta E_{5\%} = 670 \text{ cm}^{-1}$) to that determined from the emission spectra ($\Delta E = 766 \text{ cm}^{-1}$).

For optical temperature sensing applications, sensitivity (S) is an important index that reflects the performance of the obtained sensor, and the sensitivity is defined as the rate of change of the FIR with temperature:^{16–19}

$$S = \frac{\mathrm{dFIR}}{\mathrm{d}T} = \mathrm{FIR} \cdot \frac{\Delta E}{kT^2} \tag{4}$$

Fig. 8 shows the dependence of the sensitivity (S) of $Y_7O_6F_9:xEr^{3+}$ on the temperature from 300 to 550 K. The solid line is the theoretical line based on eqn (4). It is observed that a maximum sensitivity of 0.0050 K^{-1} was achieved at 498 K in the lowest Er^{3+} doped sample, and that the value was almost 1.7 times higher compared to the highest Er^{3+} doped sample.

3.4 Demonstration of the temperature measurement

To demonstrate the potential application of $Y_7O_6F_9$: Er^{3+} as a temperature sensing material, $Y_7O_6F_9$: Er^{3+} was used to determine



Fig. 9 EL spectrum of the LED coated with $Y_7O_6F_9$:1%Er³⁺ and operated at U = 3.5 V and I = 300 mA. The left upper inset is the emission spectrum of the LED, and the right upper inset photographs are the naked LED, the LED coated with $Y_7O_6F_9$:1%Er³⁺ without and with a driven current (300 mA), respectively.

the chip temperature of a 1 W InGaN type n-UV LED (epileds products; wavelength peak: 375-380 nm; chip size: 40×40 mil; forward voltage: 3.4-3.8 V). In a practical experiment, a few milligrams of Y₇O₆F₉:1%Er³⁺ was glued just on the top of the LED chip (see inset in Fig. 9). The n-UV LED chip was selected because it is can be used as either the heater or light source to excite the Y₇O₆F₉:1%Er³⁺ with the strongest absorption at 378 nm (Fig. 4a). This selection greatly simplified the experimental procedure, because if the selected LED was unable to excite $Y_7O_6F_9$:Er³⁺, then light from a spectrometer would need to be used, however, the small size of the chip makes measurement difficult. In the meantime, an appropriate electrical current must be applied to allow the LED to work. Both of these factors will make the measurement of the temperature complicated. The EL spectrum of the chip is shown in the right upper inset in Fig. 9. As the EVERFINE spectrophotometer used for the EL spectra measurement does not show a response below 380 nm, the emission spectrum of the LED was measured using a Fluorlog-3 spectrophotometer and is presented separately. Fig. 9 shows the EL spectrum of the 1 W LED coated with $Y_7O_6F_9$:1%Er³⁺ and operated at U = 3.5 V and I = 300 mA, and emission color of $Y_7O_6F_9$:1%Er³⁺ is shown in the inset. From the reference data presented in Fig. 7b and the EL spectrum shown in Fig. 9, the temperature of the chip operated at U = 3.5 V and I = 300 mA was determined to be about 95 °C.

4. Conclusions

In summary, different concentrations of Er^{3+} doped $Y_7O_6F_9$ phosphors were prepared through the phase transformation from $YF_3 \cdot 1.5NH_3$, which was obtained using the co-precipitation method, and their luminescence properties were investigated. By increasing the Er^{3+} doping concentration, the lifetime of the Er^{3+} green emission at 547 nm was significantly reduced and the temperature sensitivity of the Er^{3+} doped $Y_7O_6F_9$ decreased continuously. This was proven to be caused by the increased

cross relaxation process between two neighboring Er^{3+} *via* (Er^{3+} ${}^{4}\text{S}_{3/2} \rightarrow {}^{4}\text{I}_{13/2}:\text{Er}^{3+} {}^{4}\text{I}_{15/2} \rightarrow {}^{4}\text{I}_{11/2}$) and the increased re-absorption of Er^{3+} owing to the large spectral overlap between the emission and absorption green bands from Er^{3+} , respectively. As a proof of concept, $\text{Y}_7\text{O}_6\text{F}_9:1\%\text{Er}^{3+}$ was used to determine the temperature of a LED chip. The chip temperature of a 1 W InGaN type n-UV LED chip operating at U = 3.5 V and I = 300 mA was determined to be about 95 °C.

Conflicts of interest

There are no conflicts to declare.

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