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单晶体 α -NaYF₄ : Dy³⁺ 的制备及光谱特性

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摘 要:以氟化钾(KF)作为助熔剂,选用 70~90 °C/cm 的固液界面温度梯度,通过改进的助熔剂-布里奇曼方法生长出了 Dy³⁺ 离子掺杂的 α -NaYF₄ 单晶体.测试了样品的 X 射线粉末衍射,吸收光谱,激发光谱,以及在不同紫外光激发下的发射光谱,分析了样品的物相和光谱特性.应用色度学理论计算了 Dy³⁺ 离子掺杂 α -NaYF₄ 单晶的色度坐标和色温.结果表明:当原料组份配比为 30NaF-18KF-52YF₃ 时,有利于大尺寸晶体生长.原料中 KF 的加入起到助熔剂作用,它改变了 NaF-YF₃ 二元系相图并且降低了 α -NaYF₄ 的熔点,使得 α -NaYF₄ 能从熔融的 NaF-KF-YF₃ 混合物中结晶出来,最后生长出透明的 α -NaYF₄ 单晶体.实验发现大多 KF 被排析在生长后期的晶体顶端,KF 的加入没有对 α -NaYF₄ 单晶体的结构产生影响.在 348 nm 紫外光激发下,单晶体可发射出蓝光约 479 nm,黄光约 571 nm 以及弱的红光约 659 nm.其中 1.299 mol% Dy³⁺ 掺杂浓度的 α -NaYF₄ 单晶体发射的蓝、黄与红光可耦合出色坐标 $x=0.285$, $y=0.338$ 以及色温为 8 065 K 的白光.这些新型材料在紫外激发的白光二极管领域具有潜在的应用.

关键词:光学材料;光谱;布里奇曼方法; α -NaYF₄ 单晶体;白光发射;紫外光

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Growth and Optical Spectra of Dy³⁺ Doped α -NaYF₄ Single Crystal

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Abstract: The Dy³⁺ doped α -NaYF₄ single crystals were synthesized by a modified Bridgman method under the conditions of taking KF as an assistant flux and a temperature gradient (70~90 °C/cm) at the solid-liquid interface. X-ray diffraction, absorption spectra, excitation spectra, and emission spectra were measured to investigate the phase and spectrum properties of the crystals. The chromaticity coordinates and color temperature of crystals were calculated based on Colorimetry theories. Many growing experiments demonstrate that the chemical compositions of 30NaF-18KF-52YF₃ (molar ratio) are

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beneficial to obtain big α -NaYF₄ single crystals. The introduction of KF changed the phase equilibrium between NaF and YF₃ completely and lowered the melting point of α -NaYF₄, resulting into crystallization of α -NaYF₄ just from the mixture when it is cooled. Most of KF was excluded on the top of crystals which grew at the final stage of crystal growth. The introduction of flux KF did not change the structure of α -NaYF₄ single crystals. The emission blue 479 nm, yellow 571 nm and weak red 659 nm could be obtained simultaneously under the excitation of ultraviolet lights. A white light emission from the coupling of the above three lights with chromaticity coordinates of $x = 0.285$, $y = 0.338$, color temperature $T_c = 8065$ K could be obtained from 1.299 mole% Dy³⁺ doped α -NaYF₄ single crystal when excited by a 348 nm light. This indicates that Dy³⁺ doped α -NaYF₄ single crystals can be a potential candidate for white light emitting diode excited by ultraviolet light.

Key words: Optical material; Optical spectrum; Bridgman method; α -NaYF₄ single crystal; White light emission; Ultraviolet light

OCIS Codes: 160.4670; 160.4760; 230.3670; 260.1180

0 Introduction

Recently, the rare-earth ions doped single-host full-color emitting solid state materials for white Light Emitting Diode (w-LED) application have attracted a significantly increasing attention because they exhibit favorable characteristics such as high brightness, high luminous efficiency, low light scattering and low power consumption. These advantages could overcome the drawbacks of using the common two methods to achieve a white light emission^[1-3]. The w-LED now is an important class of lighting devices. It exhibits a high potential for replacement of conventional lighting sources such as incandescent and fluorescent lamps, and will be applied widely in the future^[1]. So far, a lot of works have been carried out on the possible application of rare earth ions doped single-host full-color emitting materials like glasses, glass ceramics for w-LED^[4-6].

As is well known, the host materials have also been playing a significant role in lights and w-LED devices. Previous work mainly focused on the glasses and glass ceramics. There are scarce reports on rare earth ions doped single crystals for w-LED due to the difficulty in crystal growth. In fact, many single crystals show a good thermal, mechanical and chemical stability, and a high luminous efficiency of rare earth ions due to their rigid cyclic symmetric structure. The excellent chemical-physical and optical properties of single crystals are favorable for them to be applied in LED device with high stability, long lifetime and high luminous efficiency. In recent years, fluoride single crystals have become the most investigated efficient luminescent material for their low phonon energy. More recently, white light emission from Tb³⁺/Sm³⁺ co-doped LiYF₄ single crystals was realized in our laboratory^[7]. Indeed, some other fluoride single crystals such as NaYF₄ existed two structures^[8-9], one being cubic α -NaYF₄ and the other hexagonal β -NaYF₄

were used as optical materials^[10-12]. Compared with other single crystals such as LiYF₄ crystals (~ 650 cm⁻¹)^[13], α -NaYF₄ single crystals have a lower phonon energy (~ 360 cm⁻¹) and a higher transmittance in UV to visible light region^[10], which are favorable for their application in w-LED.

Rare earth ions generally possess of plenty of well-shield 4f states that could emit emission covering different wavelength from ultraviolet to infrared. As a lanthanide, Dy³⁺ ions have transitions in blue region near 479 nm (⁴F_{9/2} → ⁶H_{15/2}), yellow light region near 571 nm (⁴F_{9/2} → ⁶H_{13/2}) and red light region near 659 nm (⁴F_{9/2} → ⁶H_{11/2}) when excited by a suitable ultraviolet light^[6,14-20]. These emissions including blue, yellow, and red light emitting simultaneously could potentially enable a good white light emission. It has been reported that Dy³⁺ doped solid state materials such as phosphors^[14-18], glasses^[19] and single crystals^[20] for white LED application. Compared with doubly and triply doped materials, singly doped one could reduce unnecessary energy consumption in the process of energy transfer among two or three rare earths in host materials.

In this work, Dy³⁺ doped α -NaYF₄ single crystals were obtained by an improved Bridgman method. The capability of generating white light by simultaneous emissions of red, yellow and blue emitting fluorescent centers under the excitation of ultraviolet light has been demonstrated.

1 Experiment and measurement

The Dy³⁺ doped α -NaYF₄ single crystals were successfully grown by a vertical Bridgman method. The raw materials (NaF, KF, YF₃, DyF₃ powders) with a 99.99% purity were weighed according to the molar ratio of 30NaF-18KF-51.2YF₃-0.8DyF₃ respectively. An excessive KF as a flux was critical for obtaining transparent Dy³⁺ doped α -NaYF₄ single crystals in process of crystal growth. Our experiments

demonstrated that the introduction of the fluoride salt KF changed the phase equilibrium between NaF and YF₃ completely and lowered the melting point of α -NaYF₄, resulting into crystallization of α -NaYF₄ just from the mixture when it is cooled. And then the mixture of those materials was ground about 1 h in a mortar, the moisture and some oxygen impurities in the fluoride powders could be removed completely using the high temperature hydrofluorination method by which the powders were sintered with anhydrous HF at 750 °C for 8 h. The seed from previous work was obtained by spontaneous nucleation from the seed wells and oriented along *a*-axis. The seed was put in the bottom of seed well and then the polycrystalline powders were filled in Pt crucibles with a size of $\Phi 10$ mm \times 150 mm. The temperature gradient at solid-liquid interface was 70~90 °C/cm, The growth process was carried on by lowering the crucible at a rate of ~ 0.6 mm/h. The detailed process is similar to the report of growth of LiYF₄ single crystals^[21]. The initial and middle parts of boule are transparent, while the final part of the boule is white opaque which is KF and some deviating components excluded from melt. The as-grown crystal was cut into pieces and well polished to ~ 2.0 mm thickness. One is displayed in the inset of Fig. 1.

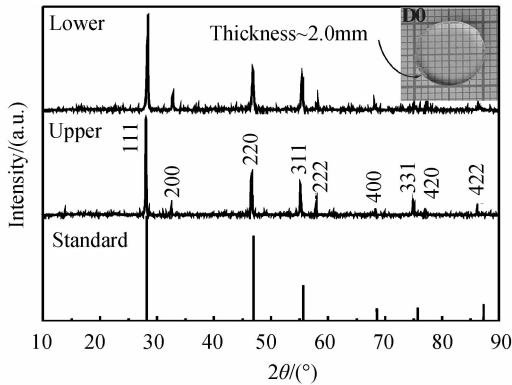


Fig. 1 The XRD pattern of the grown single crystal and standard pattern of the α -NaYF₄ (JCPD 77-2042).

The insert is the photo of polished crystal piece D₀

The Dy³⁺ doping concentrations in α -NaYF₄ single crystals were measured by the Inductively Coupled Plasma (ICP) atomic emission spectrometry (PerkinElmer Inc., Optima 3000). The 1.292 mole% and 1.299 mole% of Dy³⁺ ions were determined for the lower part of α -NaYF₄ single crystals (designated D₀), which was corresponding to the initial stage of growing process, and the upper part (designated D₁) to the final stage of growing process. A XD-98X diffractometer (XD-3, Beijing) was used to identify the phase composition of the crystal. The Cary 5000 UV/VIS/NIR spectrophotometer (Agilent Co., America) recorded the absorption spectra ranging from 240 nm to 600 nm. The optical properties of the α -NaYF₄ single

crystal doped with Dy³⁺ ions were characterized by excitation spectra and photoluminescence spectra measured on a F-4500 spectrophotometer (Hitachi high-technologies Co., Tokyo Japan). All the measurements were carried out at room temperature, and kept the same conditions for the samples in order to get comparable results. The chromaticity coordinates and color temperature of the samples were calculated from the emission spectra.

2 Results and discussion

2.1 Phase composition analysis

Figure 1 shows the XRD patterns of the lower and upper parts of obtained single crystals which are corresponding to the initial and final stages of crystal growth respectively, as well as standard card of α -NaYF₄. The visible peaks and its corresponding lattice plane indexes were also marked in Fig. 1. Comparing with reference card JCPD # 77-2042^[9,12], the diffraction peaks of both lower and upper parts of α -NaYF₄ single crystals doped with Dy³⁺ ions are well matched with those of standard α -NaYF₄, indicating that this transparent crystal has pure cubic phase. It confirms that the current Dy³⁺ ions doping level and introducing of K⁺ ions do not bring any meaningful change for single crystals structure composition. As is well known, α -NaYF₄ single crystals have fluorite structure (CaF₂), in which Na⁺ and Y³⁺ ions are randomly distributed in the cation sites where are situated in cubic center, and its lattice constants could be calculated from the XRD pattern data as following lattice parameters: $a_L = b_L = c_L = 0.5510$ nm, $a_U = b_U = c_U = 0.5562$ nm for the lower and upper part crystal respectively, which are close to the standard parameters as shown in Fig. 2.

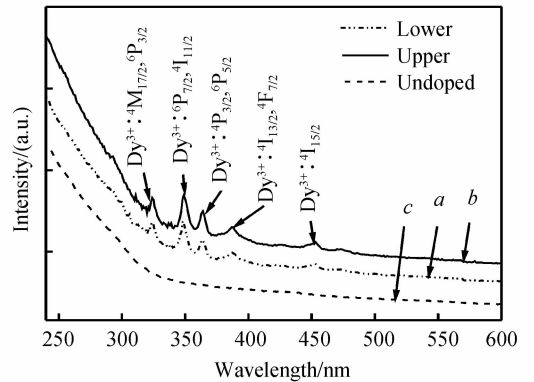


Fig. 2 Absorption spectra of the lower part, upper part and undoped α -NaYF₄ single crystals

2.2 Phase composition analysis

Fig. 2 displays the absorption spectra of the lower part, upper part and undoped α -NaYF₄ single crystals which are labeled curves *a*, *b* and *c* respectively, and

cover from 240 nm to 600 nm. It is obvious that undoped α -NaYF₄ single crystals have no visible absorption peaks referring to curve *c*. Moreover, curve *a* and *b* have the similar absorption peaks while the absorption intensity is different from each other. Five absorption peaks located at ~ 325 nm, ~ 348 nm, ~ 364 nm, ~ 387 nm and ~ 452 nm are labeled in Fig. 2, which represent the absorptive transitions of Dy³⁺ ions in α -NaYF₄ single crystal from stable state (⁶H_{15/2}) to excitation states (⁶P_{3/2}, ⁴M_{17/2}), (⁶P_{7/2}, ⁴I_{11/2}), (⁶P_{5/2}, ⁴P_{3/2}), (⁴I_{13/2}, ⁴F_{7/2}) and ⁴I_{15/2}. It is proved that Dy³⁺ doped α -NaYF₄ single crystals could be excited efficiently by ultraviolet light or blue light.

2.3 Excitation spectra analysis

The energy level transition rules could be concluded from Ref. 13 to Ref. 19 about Dy³⁺ ions and are shown in Fig. 3. It is implied that Dy³⁺ ions in the suitable host material could emit the blue, yellow and red light simultaneously under the ultraviolet excitation and that could enable a white light emission for LED application.

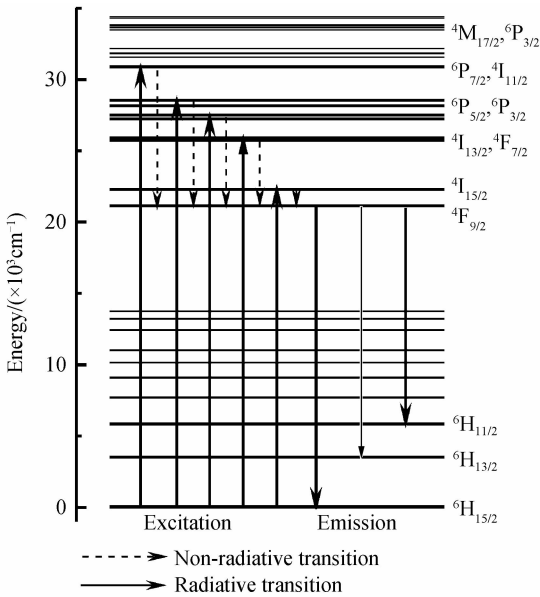


Fig. 3 The energy level diagrams of Dy³⁺ ion

To investigate the effect of excitation wavelengths on the emission spectra, we analyze the excitation spectra of the Dy³⁺ doped α -NaYF₄ single crystals. Fig. 4 exhibits the excitation spectra of the lower part crystal monitored at 479 nm, 571 nm and 659 nm which are labeled *d*, *e* and *f* respectively. There are four excitation peaks at 324 nm (⁶H_{15/2} \rightarrow ⁴M_{17/2}, ⁶P_{3/2}), 348 nm (⁶H_{15/2} \rightarrow ⁶P_{7/2}, ⁴I_{11/2}), 364 nm (⁶H_{15/2} \rightarrow ⁶P_{5/2}, ⁴P_{3/2}) and 387 nm (⁶H_{15/2} \rightarrow ⁴I_{13/2}, ⁴F_{7/2}) for the spectrum *d* and *e*. Thus, the excitation peaks at 324 nm, 348 nm, 364 nm and 387 nm are proper to be chosen as excitation lights.

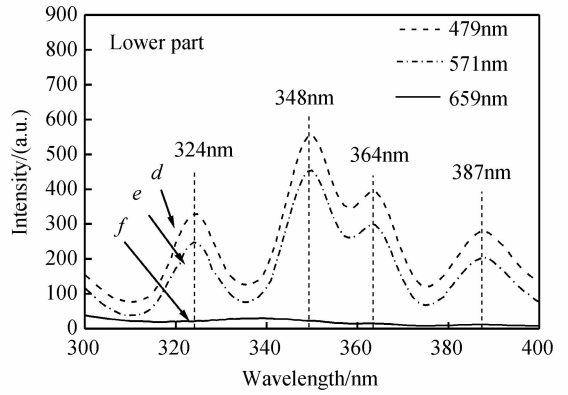


Fig. 4 The excitation spectra of the lower part crystal

2.4 Emission spectra analysis

Fig. 5 and Fig. 6 display the emission spectra of the 1.292 mole%/1.299mole% Dy³⁺ doped NaYF₄ crystal under 324, 348, 364, 387 nm wavelengths, respectively. When excited by these excitation wavelengths, the emission spectra of α -NaYF₄ single crystals doped with Dy³⁺ ions could emit a stronger blue light at ~ 479 nm, corresponding to the magnetic dipole transition (⁴F_{9/2} \rightarrow ⁶H_{15/2}), a yellow light at ~ 571 nm, related to the electric dipole transition (⁴F_{9/2} \rightarrow ⁶H_{13/2}), and a weaker red light at ~ 659 nm about the ⁴F_{9/2} \rightarrow ⁶H_{11/2} transition respectively. As is well-known, the magnetic dipole transition is insensitive to the crystal field surrounding Dy³⁺ ions while the electric dipole transition is strongly influenced by the crystal environment of Dy³⁺ ions. When Dy³⁺ ions are situated in a high symmetry site, the blue light emission would be stronger than the yellow one. In reverse, the yellow light emission is dominant in the emission^[6,13,15]. Thus, the stronger blue light emission of Dy³⁺ ions doped α -NaYF₄ single crystals from those emission spectra indicates that Dy³⁺ ions occupied a high symmetry site in α -NaYF₄ host. The emission spectra also implied that the possibility of a white light

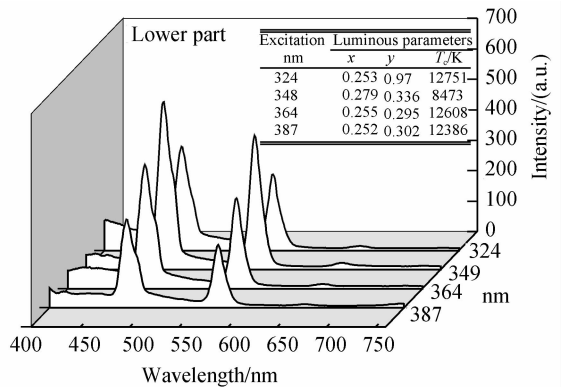


Fig. 5 The emission spectra and luminous parameters of 1.292mole% Dy³⁺ doped NaYF₄ crystal under various excitation wavelengths

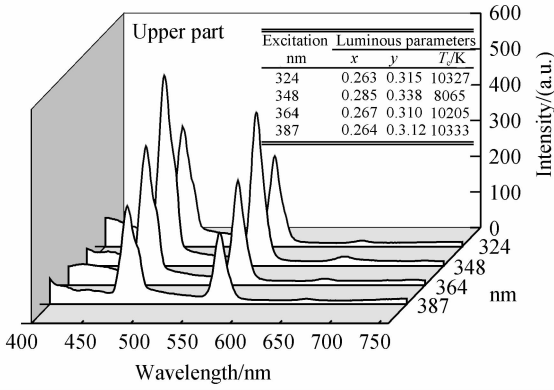


Fig. 6 The emission spectra and luminous parameters of 1.299mole% Dy³⁺ doped NaYF₄ crystal under various excitation wavelengths

emission from the combination of above blue, yellow and red lights could be come true in Dy³⁺ ions doped α -NaYF₄ single crystals.

2.5 CIE chromaticity coordinates

It is necessary to mark chromaticity coordinates of the samples on standard chromaticity diagram for reflecting their luminescence color, the chromaticity coordinates for the emissions of the Dy³⁺ doped NaYF₄ crystal under various excitation wavelengths can be calculated using the following formula^[22]

$$x = \frac{X}{X+Y+Z}, y = \frac{Y}{X+Y+Z}, z = \frac{Z}{X+Y+Z} \quad (1)$$

Where X , Y and Z are three tristimulus values. Those three values are given by the following formula^[22]

$$\begin{aligned} X &= \int_{\lambda} P(\lambda) \bar{x}(\lambda) d\lambda \\ Y &= \int_{\lambda} P(\lambda) \bar{y}(\lambda) d\lambda \\ Z &= \int_{\lambda} P(\lambda) \bar{z}(\lambda) d\lambda \end{aligned} \quad (2)$$

Where λ is the wavelength of the equivalent monochromatic light. $P(\lambda)$ is spectral power distribution. $\bar{x}(\lambda)$, $\bar{y}(\lambda)$ and $\bar{z}(\lambda)$ are the three color matching functions.

Color temperature (T_c) is also a key technical factor for evaluating the applicability of a luminescence material. Several methods for calculating T_c have been published^[23-25]. The McCamy^[23] method only provides a maximum absolute error of less than 2 degrees Kelvin for color temperatures below 5 000 K. The method of Qiu Xing-zhong^[24] which involves different equations in two ranges of an angle in u , v space, is best, but it is more complicated than former formula. In this paper, the T_c can be calculated by software (Chromaticity Coordinates To Color Temperature Conversion).

The chromaticity coordinates of the lower part, upper part crystals under various excitation wavelengths were calculated using Eq. (1) ~ (2) and they are listed in Fig. 5, Fig. 6 respectively. These

chromaticity coordinates were marked in CIE chromaticity coordinate diagrams exhibited in Fig. 7 and Fig. 8. It could be found that the emission light under a 348 nm excitation wavelength falls into the white light region. Comparing Fig. 7 with Fig. 8, we could find that the emission of the upper part crystal under a 348 nm excitation wavelength is much closer to the ideal coordinate. Although the white emission could be achieved, more effort should be paid to decrease the color temperature of α -NaYF₄ single crystals doped with Dy³⁺ ions, which is relegated to further work.

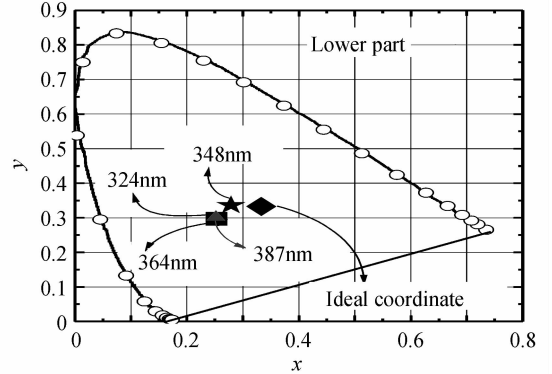


Fig. 7 CIE chromaticity coordinates for the lower part crystal under the various excitation wavelengths

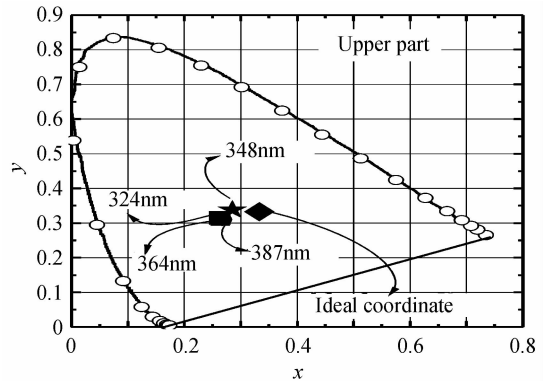


Fig. 8 CIE chromaticity coordinates for the upper part crystal under the various excitation wavelengths

3 Conclusions

As a whole, Dy³⁺ doped α -NaYF₄ single crystals could be synthesized by a modified Bridgman method. The KF was found to be a very favorable assistant flux to grow α -NaYF₄ single crystal in NaY-YF₃ system and the Dy³⁺ ion was easy to institute for Y³⁺ ion due to the same value state and similar size of radii. Strong emissions at blue \sim 479 nm, yellow \sim 571 nm and weak emission at red \sim 659 nm could be obtained simultaneously under excitation of ultraviolet lights. A white light emission from the coupling of the above three lights with chromaticity coordinates of $x=0.285$, $y=0.338$, color temperature $T_c = 8 065$ K could be obtained from 1.299 mole% Dy³⁺ doped α -NaYF₄

single crystal when excited by a 348 nm light. And the chromaticity coordinate and color temperature of the crystal was varied slightly with the excitation wavelength or adjusting the concentration of Dy^{3+} ion in $\alpha\text{-NaYF}_4$ crystals. Due to the chemical stability, high luminous efficiency and unique white light-emitting of Dy^{3+} doped $\alpha\text{-NaYF}_4$ single crystals, it can be a potential candidate for white LED application.

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