Investigation of phosphor compositions for led filament bulb

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Abstract. The paper describes the results of research and development of a phosphor composition for an LED filament. Phosphoric composition is designed to produce white-colored glow of LEDs and LED strands. It consists of both phosphor particles and a binder based on an optically transparent compound. Investigations of the chemical composition, the size of the phosphor particles, the refractive index of the phosphor compositions, the dependence of the correlated color temperature on the ambient temperature are determined. The light-emitting diodes were made on the basis of the investigated phosphor compositions, the luminous efficacy value was 193 lm/W with the correlated color temperature of 5960 K. As a result of studies of the effect of ambient temperature on phosphor compositions, it was established that on all samples of the phosphor composition the correlated color temperature rises with the ambient temperature medium from +25°C to +125°C an average of 2500 K. The results of the research extend to LEDs, LED matrixes and LED strands of white glow.

1. Introduction

Phosphors play an important role in modern lighting technology play phosphors. It is due to the phosphors that it became possible to develop highly efficient and reliable LED lighting. To get the white color of the light emitting diode, it is necessary to apply a yellow-green phosphor to the blue-colored crystal, or to apply a mixture of yellow, green and red phosphors to the crystal of purple glow [1-4]. A phosphor is an inorganic yttrium aluminum garnet activated by cerium, a powder with particles of 5-30 μm in diameter. There are four main ways of applying the phosphor to the LED crystal:

1) applying the phosphor powder directly to the crystal;
2) filling the reflector of a light-emitting diode with a crystal with a phosphor composition of phosphor particles mixed with an optically transparent compound;
3) application in two layers, on a compound crystal, then on a compound a phosphor composition;
4) phosphor is applied to an optically transparent plate and is illuminated by a blue-colored LED.

These methods have their advantages and disadvantages [2]. When manufacturing LED strings for LED lamps, the method of applying a phosphor composition to crystals is usually used. The consistency and particle size of the phosphor composition affect such parameters such parameters of the LED filament as: luminous flux, luminous efficacy, chromaticity coordinates, correlated color temperature, color rendering index and light intensity curve [5-7]. The quality of a phosphor composition and modes of its operation affect the degradation of the above parameters during operation and under the influence of external factors: climatic, mechanical, electrical, etc. [8, 9]. Thus,
the development and research of a phosphor composition is obviously an actual task in the organization of a production of LED filament and LED filament bulb on their basis.

The aim of the work is to create a high-performance phosphor composition for a LED filament. To achieve this goal, the following tasks must be solved to:

- select the components of the phosphor composition in terms of chemical composition, particle size, optical properties and lighting characteristics;
- make models of the phosphor composition and study the effect on their properties of various thermal operating modes;
- make models of LEDs for analysis of luminous efficacy.

2. Technical parameters of materials

As a binder in the phosphor composition, optically transparent silicone rubber is used, the main technical characteristics are given in table 1.

| Property                      | Unit | Value     |
|-------------------------------|------|-----------|
| Color                         | More table copya | Transparent |
| Mixing ratio                  | A:B  | 9:1       |
| Viscosity of mix              | mPa s| 800       |
| Pot life at 23 °C (up to 5,000 mPa s) | min | 90       |
| Density at 23 °C              | g/cm³| 0.96      |
| Hardness Shore A              |      | 25        |
| Dielectric strength           | kV/mm| 23        |
| Volume resistivity            | Ω cm | 1016      |
| Dielectric constant           | εr   | 2.7       |
| Dissipation factor            | tan δ| 20×10⁻⁴   |
| Tracking resistance           | CTI  | >600      |
| Temperature / Curing time, thickness 1 cm | | 23 °C 24 h; |
|                               |      | 70 °C 30 min; |
|                               |      | 100 °C 8 min; |
|                               |      | 150 °C 5 min. |

For research, three types of phosphors (type 1, type 2 and type 3) were chosen. The technical characteristics of the phosphors are given in table 2.

3. Investigation of chemical composition and particle size of the phosphors

Investigations of the chemical composition and particle size of phosphors were carried out using a TM 1000 "Tahtelop Microscope" reflective electron microscope. The chemical composition is determined by measuring the energy of X-ray radiation produced by the interaction of an electron beam with the sample surface. Micrographs of phosphor particles are obtained with a magnification of 20 to 10 000.

Micrographs of the phosphor particles are shown in figure 1. The chemical composition of phosphors is shown in figure 2 in atomic percent. As a result of the conducted researches it is established:

- type 1 of phosphor has a characteristic particle diameter of 10 μm, an atomic percentage chemical composition of Y-35%; Al-52.7%; Rh-9.9%; Ir-2.5%;
- type 2 of phosphor has a characteristic particle diameter of 15 μm, an atomic percentage chemical composition of O-40.7%; C-43.3%; Y-5.3%; Al-7.3%; N-3.3%;
- type 3 of phosphor has a characteristic particle diameter of 15.5 $\mu$m, an atomic percentage chemical composition of O-40.3%; Y-5.7%; F-1.1%; Al-14%; C-39.9%.

**Table 2.** The main technical characteristics of the phosphors.

| Property                                | Unit     | Value     |
|-----------------------------------------|----------|-----------|
| **Type 1**                              |          |           |
| Form                                    |          | yellow-green powder |
| Particle size                           | $\mu$m   | 6–20      |
| Relative intensity                      | %        | 100       |
| Maximum of the radiation spectrum       | nm       | 538–542   |
| **Type 2**                              |          |           |
| Form                                    |          | yellow-green powder |
| Particle size                           | $\mu$m   | 10–26     |
| Relative intensity                      | %        | 98        |
| Maximum of the radiation spectrum       | nm       | 554       |
| **Type 3**                              |          |           |
| Form                                    |          | yellow powder |
| Particle size                           | $\mu$m   | 10–26     |
| Relative intensity                      | %        | 98        |
| Maximum of the radiation spectrum       | nm       | 560       |

**Figure 1.** Micrographs of the phosphor particles: a – type 1, b – type 2, c – type 3.
4. Research of optical properties and colorimetric characteristics of the phosphor compositions
Samples of the phosphor compositions 1 mm thick in the form of plates on quartz glass 2 mm thick.
- Plate of compound without phosphor.
- Sample 1–7% of phosphor type 1 in the total mass of the compound.
- Sample 2–7% of phosphor type 2 in the total mass of the compound.
- Sample 3–7% of phosphor type 3 in the total mass of the compound.

4.1 Investigation of optical properties
We carried out a study of the refractive index, the absorption coefficient and the reflectance of the compound and samples of phosphor compositions by the laser ellipsometer PHE-101. The results of the studies are shown in table 3.
Table 3. Results of study of the refraction indicator, absorption coefficient and reflection indicator.

| Type     | The refractive index, (n) | The absorption coefficient, (k) | Reflection in % |
|----------|--------------------------|-------------------------------|-----------------|
| Compound | 1.367                    | 0.03                          | Max 8%          |
| Type 1   | 1.529                    | 0.023                         | Max 1.4%        |
| Type 2   | 1.29                     | 0.025                         | Max 5.5%        |
| Type 3   | 1.618                    | 0.045                         | Max 4.3%        |

4.2 Research of colorimetric characteristics

We carried out investigations of the emission spectra of samples of phosphor compositions as a function of the ambient temperature using a spectrometer. During the experiment samples of phosphor compositions were heated and irradiated with a non-heated blue-colored LED with a peak wavelength of 460 nm. The correlated color temperature is calculated from the obtained data. The results of the studies are shown in figure 3.

Figure 3 shows that with increasing ambient temperature, the correlated color temperature increases. Moreover, in the area of dependence, in the range from 25 to 85°C, linear growth is observed on average in the range of 700 K, then as a result of temperature quenching in the area from 85 to 100°C. This increase is amplified and fully manifested in the area from 100 to 120°C. On average, in the temperature range from 25 to 120 °C, the growth of the correlated color temperature is 2150±500 K.

5. Manufacture and investigation of led lines based on the developed the phosphor compositions

Models of light-emitting diodes are made for definition of lighting technical characteristics of phosphor compositions. The crystals are selected of medium power, which is used in both LEDs and LED filaments. The external appearance and overall dimensions of the crystal are shown in figure 4.

![Figure 3](image1.png)

**Figure 3.** Dependence of the correlated color temperature of samples of the phosphor compositions on the ambient temperature.

![Figure 4](image2.png)

**Figure 4.** Dimensional drawing of the crystal.

The crystal is mounted with a heat-conducting adhesive in the SMD 5050 LED housing. The contacts of the crystal and the housing are connected by means of ultrasonic verification using metal wire. Made models of LEDs:

- Procurement of a light-emitting diode without the phosphor composition.
• Model of LED 1-7% of phosphor type 1 in the total mass of the compound.
• Model of LED 2-7% of phosphor type 2 in the total mass of the compound.
• Model of LED 3-7% of phosphor type 3 in the total mass of the compound.

With the help of an integrating sphere, a spectrometer and a source-meter of electrical characteristics, studies of the characteristics of LED dummy's have been carried out. The measurement results are shown in table 4. The measurements were carried out under normal conditions.

| Type        | Luminous flux, F [lm] | Luminous efficacy, η [lm/W] | Correlated color temperature, CCT [K] |
|-------------|-----------------------|-----------------------------|---------------------------------------|
| LED         | 0.75                  | 26                          | –                                     |
| Model of LED 1 | 5.2                  | 179                         | 5839                                  |
| Model of LED 2 | 8.5                  | 193                         | 5960                                  |
| Model of LED 3 | 8.4                  | 189                         | 4675                                  |

6. Conclusion
An experimental study was made of the chemical composition and particle size of the three phosphors. Using phosphors and an optically transparent silicone compound, samples of phosphor compositions 1 mm thick in the form of plates on quartz glass 2 mm thick were made. Investigations of the refractive index, the absorption coefficient and the reflection index of the compound and three samples of phosphor compositions were carried out. The emission spectra of phosphor compositions were studied as a function of temperature. During the experiment samples of phosphor compositions were heated and irradiated with a non-heated blue-colored LED with a peak wavelength of 460 nm per lumen. As the temperature of the phosphor compositions increases, the correlated color temperature of the LED and phosphor radiation increases. In the temperature range from 25 to 85°C linear growth is observed on the average by 700 K. With the use of three phosphor compositions, models of light-emitting diodes are made. The value of the luminous efficacy is 193 lm/W with a correlated color temperature of 5960 K. The results of the research can be used in the design of super-bright LEDs, LED matrices and LED threads for white-colored lamps.

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