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# The preparation of GaN:Er<sup>3+</sup> nanoparticles and the optical performance analysis

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#### A R T I C L E I N F O

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#### ABSTRACT

GaN:Er<sup>3+</sup> nanoparticles are synthesized by ammonification of a mixture of Ga<sub>2</sub>O<sub>3</sub> and Er<sub>2</sub>O<sub>3</sub> in three steps. The XRD result shows that three groups of samples predominantly presents hexagonal phase of GaN and its average grain size is 25.9 nm. SEM images show that the sizes of the particles are almost uniform. The sizes of the particles is 20–50 nm. The optic characteristics of the samples is tested in 380 nm photoluminescence(PL). From PL spectra, three characteristic peaks of Er<sup>3+</sup>ions are clearly observed in visible light region (375–650 nm), this prove the GaN:Er<sup>3+</sup> nanoparticles possess good optical properties. Simultaneously, analyze the three samples in the 520 nm excitation spectrum. Among them, the sample of the doping molar concentration 6.25% achieved the most excellent optical properties.

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Gallium nitride semiconductor materials, namely GaN, are the third generation semiconductor materials after the first generation of represented by Si and the second generation of represented by GaAs.Group III-nitrides, including binary AlN, InN, GaN and ternary InGaAs, AlGaAs and quaternary InGaAlAs etc. are obtained band gap of ranging from 1.89 eV(InN) to 3.39 eV (GaN), to 6.2 eV (AIN) by adjusting the alloys components, correspondingly, the light wavelength range from 200 nm to 656 nm. Because of these reasons, Group III-nitrides represented by GaN are highly promising material systems in short blue wavelength range and violet light and UV light emission devices [1–3].

Because the rare earth element compounds mostly consist of RE<sup>3+</sup> ions, trivalent rare earth ion RE<sup>3+</sup> doping GaN generates a replacing doping accompanying the location of the Ga in GaN lattice. Thus, it can improve transition probability in electronic shell of 4f, it is also very useful to improve the luminous efficiency. Rare earth ion luminescence is mostly the electron transition fluorescence in electronic shell of 4f. Rare earth ion's transition is minimally influenced by the outside world, inner electrons are shielded by the outer 5s5p electronic shell. The location of the rare earth ion's photoluminescence peak is nethier basically affected by the substrate material, nor depends on the intensity of excitation light and environment conditions. The rare earth ion's photoluminescence peak of free rare earth ions. Theoretical studies have shown that, the

http://dx.doi.org/10.1016/j.ijleo.2016.01.159 0030-4026/© 2016 Elsevier GmbH. All rights reserved. more semiconductor materials' band gap is wider, the more semiconductor materials' temperature of quenching effect is lower. For these reasons, GaN has become one of the ideal substrate materials of RE<sup>3+</sup> ions doped luminescence.[4–6] In order to seek a simple wet chemical route to synthesize GaN of rare-earth-doped, the research group has tried to employ three steps of oil bath heating in magnetic agitator, high-temperature calcination, and ammonification method. Very few people ever tried this method. The research group studied on its optical properties of rare earth ions doped concentration, obtained some valuable results.

#### 1. Experimental

#### 1.1. Materials

The experiment used Ga<sub>2</sub>O<sub>3</sub>(99.99%) and the concentrated nitric acid(the purity of 99.99%) and Er<sub>2</sub>O<sub>3</sub> (99.99%;particle size is 200) as raw material samples. According to the target product the mole ratio of Er:Ga 2%, 4%, 6.25%, respectively, calculated the required amount of raw materials in the preparation of GaN:Er<sup>3+</sup> nanoparticles in three groups of samples. According to the proportion of Er doped, mixed Ga<sub>2</sub>O<sub>3</sub> and Er<sub>2</sub>O<sub>3</sub> accordingly with 25 ml, 30 ml, 35 ml nitric acid; then synthesized GaN:Er<sup>3+</sup> nanoparticles in three steps.

#### 1.2. Synthesis GaN:Er<sup>3+</sup> nanoparticles

#### 1.2.1. Magnetic stirring and oil bath heating process

Put the mixed solution of  $Ga_2O_3$   $Er_2O_3$  and  $HNO_3$  into the heating type magnetic agitator, set the oil bath heating  $120 \degree C$ , 60 min,







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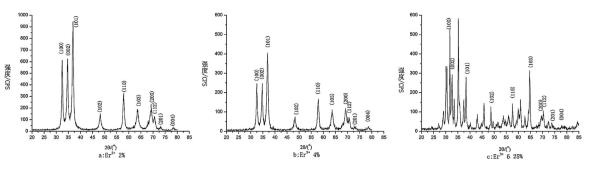


Fig. 1. XRD patterns of three different doping ratio of GaN:Er3+ nanoparticles.

magneton speed 30 r/min, make them stirred and dried, then get the nitrate containing water of crystallization.

#### 1.2.2. High temperature calcinating process

Put the nitrate into quartz tube furnace and calcined, calcine nitrate 240 min at the temperature of 500 °C, cooled to room temperature, get the precursor materials – the oxide including Er and Ga.

*Note:* Both ends of the quartz tube in this process is through the air.

#### 1.2.3. High temperature ammoniating process

Get the precursor material grinding for 60 min in the agate mortar, then put the precursor material into quartz tube furnace, set procedures to ammonification, as follows.

#### 1.3. Characterization

The samples of expected for GaN:Er<sup>3+</sup> nanoparticles are faint yellow granular. Then, the crystal structure and phase purity of the as-synthesized GaN nanopowder samples are characterized by Xray diffraction(D8 Advance) with Cu  $K_{\alpha}$  radiation( $\lambda = 0.15406$  nm). On the basis of the Scherrer formula, obtained average grain size of the GaN:Er<sup>3+</sup> nanoparticles. The surface morphology is studied by using field emission scanning electron microscopy (JSM-7001F FESEM). Optical characteristics of the GaN:Er<sup>3+</sup> nanoparticles are mainly recorded using the PL spectra(Hitachi F-7000). Room temperature photoluminescence spectra (PL) and excitation spectrum (PLE) of the samples are detailed analysis, with 380 nm Xe lamp (300 W) as the excitation source, excitation wavelength range from 300 nm to 700 nm.

#### 2. Results and discussion

#### 2.1. The XRD analysis of the samples

The structure of the GaN: $Er^{3+}$  nanoparticles is characterized by XRD first. Fig. 1(a)–(c) showed the three X-ray diffraction patterns of samples at the temperature of 950 °C respectively. Among them the graph(a) is a X-ray spectra of the erbium-doped (Er) 2%; the graph(b) is a X-ray spectra of the erbium-doped (Er)4%; the graph(c) is a X-ray spectra of the erbium-doped (Er) 6.25%.

For graph(a) and graph(b), all the diffraction peaks are indexed and identified as the hexagonal wurtzite structure of GaN, which agree well with the JCPDS values of GaN hexagonal structure(card no: 65-3410). Spectral line was sharp and narrow, this presented the samples are a better six-party phase crystalline of GaN.No other crystalline phases associated with rare earth Er and their compounds. This showed rare earth  $Er^{3+}$  ions did not change the crystal structure of GaN because rare earth  $Er^{3+}$  ions' doping ratio is too low, they have been evenly mixed into the GaN substrate. For graph(c), some different diffraction peak can be seen different from GaN standard spectrum. Using the XRD analysis software, further analysis, standard peak of GaN was found, in addition the obvious diffraction peak of erbium gallium nitride(JCPDS card no: 12-0769) can be seen, and besides, contains a small amount diffraction peak of erbium gallium oxide. Meanwhile, the diffraction peaks intensity of graph(c) is slightly increased than graph(a) and graph(b), and the diffraction peak narrowing, the full width at half maximum(FWHM) decreasing. These shows that the crystallization degree of crystalline grain strengthen and the grain size increases with the change of the doping concentration. JCPDS card is used to confirm the X ray peaks belonging to which phase. It is found that three groups of samples identified index of crystallographic plane of diffraction peaks is belonged to six-party GaN (h-GaN), this coincided exactly with the standard diffraction peaks of the GaN in the JCPDS card no: 65-3410. Three peaks of three groups of samples were both located in  $2\theta$  = 32.410°, 34.613°, 36.894°. Which correspond to the plane diffraction (100), (002) and (101) of GaN. From the diffraction pattern, it can be seen a few of weak diffraction peaks belong to h-GaN, which peak position distribute in  $2\theta = 48.183^{\circ}$ , 57.848°, 63.531°, 67.861°, 69.175°, 70.606°, 73.080°, this correspond to crystallographic plane (102), (110), (103), (200), (112), (201), (004) of h-GaN, respectively. That means the doped rare earth Er<sup>3+</sup> ions may occupy lattice position or interstitial position of the Ga<sup>3+</sup> ions. But it does not change the structure of the GaN substrate characteristics. The particle size (D) of the GaN:Er<sup>3+</sup> nanoparticles is calculated from the XRD peaks using Scherrer's formula: [1,2]

$$D(nm) = \frac{K\lambda}{\beta\cos\theta}$$

where D is the diameter of the crystalline material; K is a constant which depends on the crystalline shape, the value of it is 0.89;  $\lambda$  is the wavelength of incident X-rays and  $\beta$  is the full width at half maximum(FWHM) and  $\theta$  is the Bragg angle. Computed the three peak of (100), (002) and (101), and then averaged. The calculation of three groups of sample indicate that the average crystalline grain size of GaN:Er<sup>3+</sup> nanoparticles (D) is 23.07 nm, 29.13 nm, 25.56 nm, respectively; with a average value of 25.92 nm. Meanwhile, calculated the lattice constant average of GaN:Er<sup>3+</sup> nanoparticles:  $\bar{a} = 3.18766 \text{ Å}$ ,  $\bar{c} = 5.17865 \text{ Å}$ . Compared with the JCPDS card, these calculations are a little bit larger than the diffraction results of GaN powder standard: No. 65-3410, its standard value is: a = 3.186 Å, c = 5.176 Å. Analysis of the reason: it is considered that may be rare earth Er<sup>3+</sup> ions radius is larger than the Ga atomic radius, but the location of Ga in GaN is replaced by rare earth Er<sup>3+</sup> ions [5,6].

#### 2.2. The SEM analysis of the samples

Further studied the size of the GaN: $Er^{3+}$  nanoparticles by using SEM. Fig. 2(a)–(c) showed the SEM photos of three groups of samples. Results show that the size distribution of the GaN: $Er^{3+}$ 

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