The Manufacture of GaN Powder in Plasma

Yafeng Li and Wenzhi Yang

1 Zhejiang Business Technology Institute, Ningbo, 315103, China
2 Ningbo Branch of China Academy of Ordnance Science, Ningbo, 315103, China

*Corresponding author email: 10822025@zjbti.net.cn

Abstract. In this paper, the preparation technology of GaN powder has been studied by using of plasma powder synthesis system, and several properties were improved such as: the morphology, the particle size, structure and optical properties. The result shows that, by applying for plasma method, high purity GaN powder was manufactured; the GaN has a good morphology and a regular spherical shape. The spherical ratio of the GaN powder was more than 95%; The GaN powder can be adjusted with different process parameters to achieve particle size control; The GaN powder has sharp diffraction peak and good crystallinity; The GaN powder has the structure of wurtzite and good luminescent properties.

Keywords: Plasma method; GaN powder; Nano-powder.

1. Introduction
The GaN have several outstanding physical properties, such as: wide & direct band gap (3.39ev), strong atomic bond, good chemical stability (almost no acid corrosion), high thermal conductivity and strong radiation resistance. It has broad prospects in the application of optoelectronics, high-temperature high-power devices and high-frequency microwave devices[1-2].

The GaN nanopowder was a kind of basic raw material for electronics industry. But, there were several questions in the manufacture of GaN, such as low purity, low output, high price, small production scale, uneven particle size, and difficult to control product quality, it has greatly restricted the development of new electronic industry and other related industries in the world. It is urgent to develop a new and efficient GaN manufacture method[3-6].

Plasma technology was an ideal method for high energy power synthesis. In this process, the raw mixed powder was melting and vaporization because of the plasma’s high energy and the powder will become regularly[7-10]. The plasma manufacture powder technology has many advantages, such as :
(1) It can change the powder’s morphology, from irregular to sphere in microstructure The powder’s spherical ratio will reach to 95% after disposed. The treated powder is standard spherical;
(2) It can reduce the defects in the powder particles;
(3) It can improve compaction density of the powder;
(4) It can reduce the impurities content because of introducing a reducing atmosphere in the preparation process.

In this paper, we can manufacture the GaN powder using the plasma method, and study the influence of process parameters on material properties. The properties include morphology, phase, structure, optical performance.

2. Experiment
The figure 1 was schematic diagram of induction plasma powder system.
Figure 1. Schematic diagram of induction plasma powder system

The raw material (GaO powder) was sent to the plasma zone, and it will instant gasification in nitrogen atmosphere plasma. In this step, the nitrogen ions will replace oxygen ions to form GaN droplets because of high temperature role. The GaN droplets will move with the gas follows, and the high speed quench gas makes Gan drop solidify instantly. At last, these GaN nano-powder were move into glove-box.

The parameters of experiment were shown on the table:

| Type            | Parameters                                                                 |
|-----------------|----------------------------------------------------------------------------|
| Raw material    | GaO powder, which the particle size range in5-30um                        |
| Plasma gas      | Ar and H₂                                                                  |
| Quench gas      | Ar                                                                         |
| Synthesis gas   | NH₃                                                                        |
| Plasma power    | 45-60KW                                                                   |
| Powder feeding rate | 100-300g/h                     |

The GaN powder was collected from the bottom of the reactor and glove-box. We can get the spherical GaN powder’s properties from testing of SEM, BET, XRD and Raman spectrum test[11-13].

3. Results and Discussion

3.1. The Morphology

We can compare the morphology between the raw material and treated powder in below pictures.

Figure 2. SEM of GaO and GaN powder
It can be seen from the figure that the raw material powder without treatment was in irregular shape, and the particle size of the powder was between 5-20μm. After plasma treatment, the powder was regular spherical with uniform particle size distribution, and the particle size was about 200nm. Only a few particles in the statistical field of view were non-spherical, and the spherical ratio was more than 95%.

3.2. Particle Size Control

The specific surface area of powder was tested by BET, and converted into powder diameter as follows:

| Feed rate (g/h) | BET (m²/g) | Particle size d(nm) |
|----------------|-----------|---------------------|
| 50             | 11.8341   | 83                  |
| 100            | 7.4996    | 131                 |
| 200            | 3.9670    | 248                 |

With the increase of powder feeding rate, the average particle size of the powder increases. The reasons are as follows: plasma treatment process can be regarded as a high temperature, high viscosity liquid, and the cold raw material powder will bring two aspects of influence. On the one hand, the entry of cold raw material powder will significantly reduce the temperature in the plasma area, thus reducing the energy coupling degree between the plasma and raw material powder, that was, reducing the gasification rate or degree of raw material powder, further affecting the chemical reaction rate, resulting in weak chemical reaction. On the other hand, the entry of cold raw material powder will reduce the material flow speed in the plasma area, so that the raw material powder stays in the plasma area for a longer time and absorbs more energy. It can be seen that the effect of powder feeding rate on the treatment effect of plasma powder technology was a competitive mechanism. However, in the range of powder feeding rate in this experiment, it was obvious that the factors that reduce the plasma temperature play a leading role, that was, with the increase of powder feeding rate, the plasma temperature drops obviously, which can not make the raw material powder fully react, so that with the increase of powder feeding rate, the particle size of nano powder increases obviously, and even unreacted raw material powder appears.

3.3. XRD Test

Figure 3 shows the XRD pattern of GaN powder prepared by inductively plasma method. Through XRD analysis, we can see that the single-phase GaN nano-powder was obtained without other phases. The crystal structure was hexagonal structure, the space group was p63mc (186), the cell parameter was a = b = 3.189nm, C = 5.186nm, and the crystal surface index was shown in the figure. In addition, the peak of powder diffraction was sharp, which indicates that the crystallinity of GaN nano-powder was better.

3.4. Raman Spectrum Test

Figure 4 shows the Raman spectrum of GaN powder prepared by inductively plasma method. It can be seen from the figure that there are obvious Raman vibration peaks in 567cm⁻¹ and 531cm⁻¹. The peak of 567cm⁻¹ belongs to E₂ vibration mode of GaN crystal and the peak of 531cm⁻¹ belongs to A₁ (TO)
vibration mode of GaN crystal. It also proves that the GaN powder has good crystallinity, which was consistent with XRD analysis.

Figure 4. Raman pattern of GaN powder

Figure 5. Photoluminescence spectrum of GaN powder

Figure 5 shows the photoluminescence spectrum of GaN powder prepared by inductively plasma method. The photoluminescence performance of GaN nano-powder was tested by fluorescence spectrometer, in which the light source was 450W xenon lamp without ozone, and the excitation wavelength was 340nm. It can be seen from the figure that there was a sharp emission peak at 370nm, which was caused by band edge emission of GaN powder, which was very close to the emission peak at 365nm displayed by GaN film. It was also proved that the GaN powder has wurtzite structure. In addition, there was a very broad emission peak at 560nm, which was related to the defects in the material.

4. Conclusion
(1) High purity GaN powder was prepared by inductively plasma method;
(2) The GaN powder has regular spherical shape. The spherical ratio of the Gan powder was more than 95%;
(3) The GaN powder can be adjusted with different process parameters to achieve particle size control;
(4) The GaN powder has sharp diffraction peak and good crystallinity;
(5) The GaN powder has the structure of wurtzite and good luminescent properties.

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