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Synthesis and Characterization of Nd:$Y_3Ga_5O_{12}$ Nanopowder for Transparent Ceramic

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Nd:$Y_3Ga_5O_{12}$(Nd:YGG) nanopowder was prepared by ammonium hydrogen carbonate co-precipitation method. The X-ray diffraction suggested that the powder became pure phase without impurities after calcined at 900 °C for 4 h. Infrared spectrum indicated that after calcined at 900 °C, the acid radical and hydroxide are all decomposed. SEM showed that the powder reveal good dispersity with the average grain size about 60 nm. Fluorescence spectra showed that the strongest emission peak located at 1062 nm, which is the transmission of $^4F_{3/2}$-$^4I_{11/2}$ by Nd^{3+} ion.

Keywords: Nd:YGG nanopowder, XRD, Fluorescence spectra.

INTRODUCTION

Yittrium gallium garnet (YGG) is an excellent functional material with good optical and mechanical properties. Active ions doped yittrium gallium garnet can also be used as excellent laser materials. It can generate different wavelength laser with corresponding ions. Owing to the outstanding advantages, such as a long storage lifetime, a low quantum defect, a broad absorption bandwidth and less sensitivity to diode wavelength specifications, single crystals of Nd^{3+}-doped yittrium gallium garnet has been a potential solid state laser material[1-4].

As a kind of laser material, it is often use laser crystal for application. But single crystal has some restrictions, such as high-cost, low doping concentration and small size *etc*[5-7]. Comparing with single crystal, transparent ceramics become a hot research field because of its characteristics of short producing cycle, low cost, high productivity and transparent ceramics also possesses uniform optical property and high doping rate[8-10].

In this paper, Nd:$Y_3Ga_5O_{12}$ nanopowder was prepared by ammonium hydrogen carbonate co-precipitation method and the spectral properties of nanopowder were investigated. The research work will play a significant role in searching for appropriate solid laser host materials.

EXPERIMENTAL

99.999 % (5N) Ga_2O_3, 99.99 % (4N)Y_2O_3 and 99.99 % (4N) Nd_2O_3 were applied as raw materials with the Nd^{3+} concentration of 3, 4 and 5 at. %. First Ga_2O_3 and Y_2O_3 were weighted according to stoichiometric proportion 3:5. Extra 2 wt % Ga_2O_3 was added for the volatilization of Ga^{3+}. Then Ga_2O_3, Y_2O_3 and Nd_2O_3 were dissolved separately in HNO_3 with the concentration of 5 mol/L. Since Ga_2O_3 is poorly soluble in acid, right amount of hydrochloric acid was added into the Ga_2O_3 solution. The reaction equation was as follows:

$$3_xNd_2O_3 + 5Ga_2O_3 + (3\text{-}3x)Y_2O_3 \rightarrow 2Nd_{3x}:Y_{(3\text{-}3x)}Ga_5O_{12} \quad (1)$$

The acquired solutions were placed at a magnetic stirring apparatus with the temperature of 50 °C, then heated and stirred continuously. After the above solutions get clear, they were mixed sufficiently to obtain settled uniform solution, which is the mother solution. Then weighted ammonium hydrogen carbonate properly and dissolved in deionized water. In this paper, 1.5 mol/L ammonium hydrogen carbonate was chosen as precipitant. Then ammonium hydrogen carbonate was added slowly to the mother solution to get the precipitate.The pH value in the precipitant was selected as 8.5 for the complete precipitation of the solution. The precipitate was ageing for 24 h, then leaching, washing and drying to obtain the precursor. The obtained precursor was placed into muffle furnace and sintered for 4 h, the temperature were setting at 800, 900 and 1000 °C, respectively.

Rigaku D/max-II B-type X-ray diffractometer was adopted to analyze the phase structure of powder with Cu taget $K_{\alpha 1}$ radiation and graphite monochromator to filtering. The working current, working voltage, scanning space and step length were 20 mA, 40 kV, 4°/min and 0.02°, repectively.

FTS135 Fourier transform IR spectrograph was applied to analyze the IR spectrum. The resolution ratio was 4 cm^{-1} and the spectrum range were 4000-400 cm^{-1}. The morphology of powder was investigated by FESEM JSM-6700F high resolution scanning electron microscope (SEM). PL9000 photoluminescence fluorescence spectroscopy was used to test the spectral properties of the powder with the excitation wavelength of 488 nm.

RESULTS AND DISCUSSION

XRD analysis: Fig. 1. shows the XRD pattern of Nd:YGG precursor powder calcined from 800, 900 and 1000 °C for 4 h, respectively. After comparing with JCPDS standard card (43-0512), it can be seen that Nd:YGG belongs to cubic system and garnet structure with the space Ia3d, which is the same construction as pure yittrium gallium garnet. Moreover, it can be observed in the figure that the intensity of diffraction peaks become stronger and sharper along with the raise of temperature,which cound stated clearly that the crystalline of GGG increases with the increase of calcining temperature.

According to diffraction formula:

$$2d \sin(\theta) = n\lambda \quad (2)$$

Through the cubic relationship, we calculated calculation formula of lattice constants of cubic system.

$$d = \frac{1}{\sqrt{\frac{h^2 + k^2 + l^2}{a^2}}} \quad (3)$$

According to formula (2), we can obtain d values and we select four different crystal plane (h k l) values, such as: (111), (121), (200) and (002), put the corresponding d values into the formula (3) and provides four equations, solve equations for a lattice constant: a = b = c = 12.268 °C. The results were compared with the pure yittrium gallium garnet phase, which have little distinction. It shows that with the doping concentration increases, the yittrium gallium garnet phase can be pure and with low impurities.

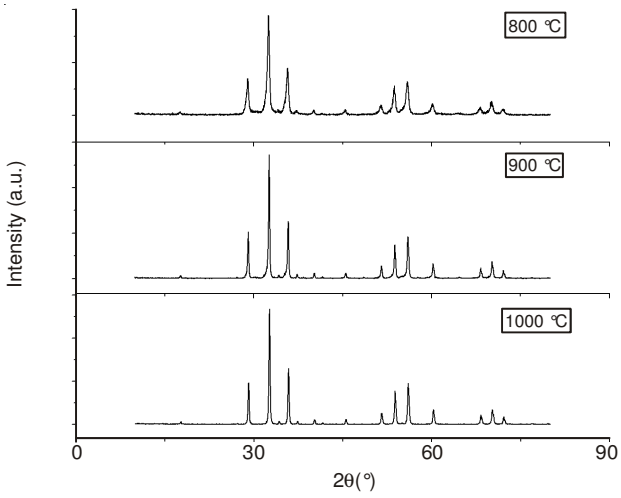

Fig. 1. XRD pattern of samples sintered at 800, 900 and 1000 °C

IR analysis: Figs. 2 and 3 depict the IR spectra of the presoma and the powder sintered at 900 °C. It can be seen in the spectra that before sintering, there are two absorption peaks

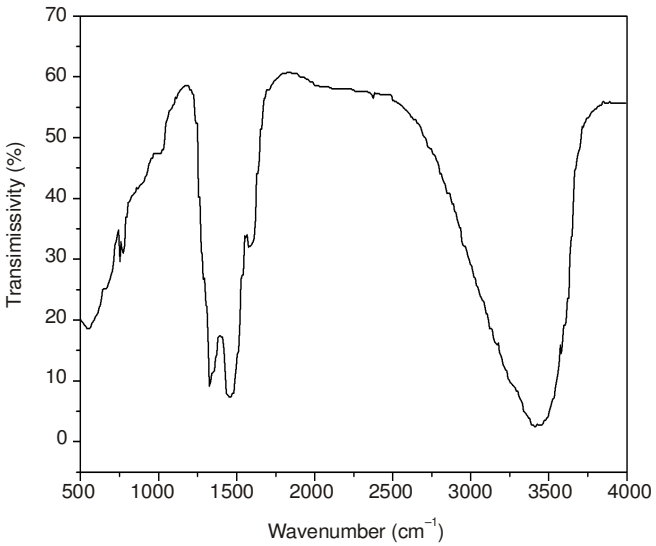

Fig. 2. IR spectrum of the presoma

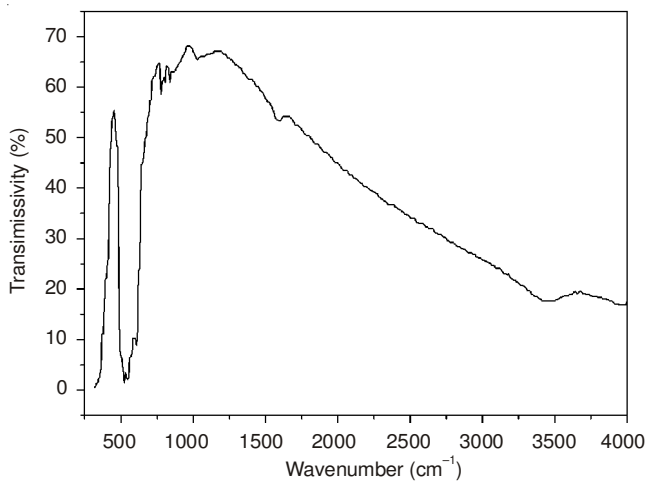

Fig. 3. IR spectrum after sintered at 900 °C

at 1500 and 3500 cm^{-1}, which caused by carbonate, hydroxyl and absorbed water. After sintering, all the peaks disappear, which indicated that all the acid radical and hydroxide were completely decomposed.

SEM analysis: Figs. 4 and 5 show the SEM images of Nd:YGG precursor powder calcined at 900 and 1000 °C for

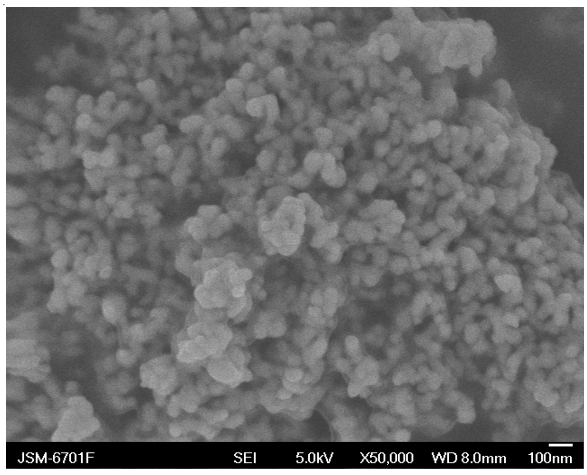

Fig. 4. Powder calcined at 900 °C

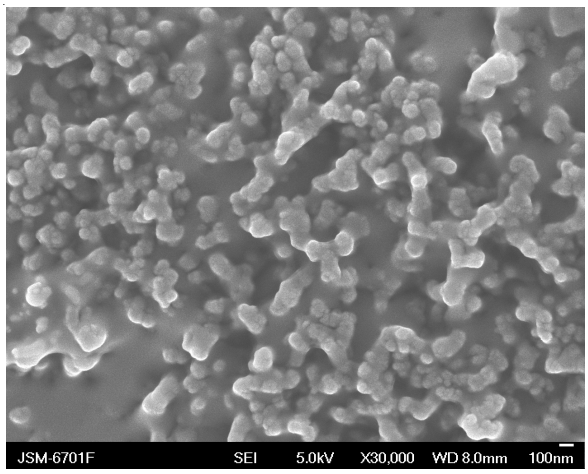

Fig. 5. Powder calcined at 1000 °C

4 h, respectively. It can be seen in the images that the powder calcined at 900 °C for 4 h reveal good dispersity with the grain size about 60 nm, which can be favourable raw materials for transparent ceramics. When calcined at 1000 °C the grain become larger with the grain size more than 100 nm, which is diffcult for preparing transparent ceramics. The surface morphorlogy of the Nd:YGG powder shows that when the calcined temperature is higher, the grain size become larger. So it is important for better controlling the calcined temperature and the holding time.

Fig. 6 shows the fluorescence spectra of the samples sintered at 900 °C for 4 h in the wavelength range from 600-1800 nm, the concentration of which are 3, 4 and 5 at.%, respectively. The intensity of emission peaks become stronger along with the increase of Nd^{3+} doping concentration at certain range, with a little peak change at the same time. There are strong emission peaks located at 1062, 1108 and 1332 nm. The main peak is located at 1062 nm, which caused by the $^4F_{3/2}$-$^4I_{11/2}$ energy level transition of Nd^{3+} ions.

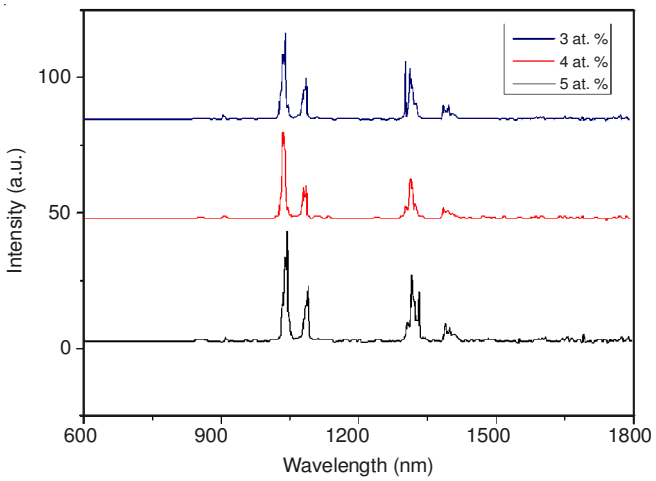

Fig. 6. Nd:YGG fluorescence spectra of different concentration

Conclusion

In this paper, Nd:$Y_3Ga_5O_{12}$ (Nd:YGG) nanopowder was prepared by ammonium hydrogen carbonate co-precipitation method. The as synthesized powder was tested by XRD, IR spectrum, SEM and fluorescence spectrum. Results showed that the powder became pure phase without impurities after calcined at 900 °C for 4 h. The lattice constant was calculated, which showed that with the doping concentration increases, the yittrium gallium garnet phase can be pure and with low impurities. IR analysis indicated that after calcined at 900 °C, the acid radical and hydroxide are all decomposed. SEM showed that the powder reveal good dispersity with the average grain size about 60 nm. The surface morphology of the Nd:YGG powder shows that when the calcined temperature is higher, the grain size become larger. The fluorescence spectrum showed that the strongest emission peak located at 1062 nm, which is the transmission of $^4F_{3/2}$-$^4I_{11/2}$ by Nd^{3+} ion. The as prepared powder can be favourable raw materials for transparent ceramics.

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