

# Preparation of Nd:YAG Powder by sol-gel Method in Comparing with Solid State Reaction.

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*Abstract:* - Nd:YAG powder was synthesized by sol-gel method and compared with solid state reaction method. The XRD patterns of the heated powders at 900 °C showed the single phase of YAG is formed. By SEM, micrographs of the synthesized samples and also homogeneity of Nd distribution in the YAG lattice was observed.

*Key words:* - laser ceramics, Nd:YAG, sol-gel synthesise, solid state lasers, Nd:YAG powder, polycrystalline Nd: YAG.

## 1 Introduction

Nd:YAG (Nd doped  $Y_3Al_5O_{12}$ ) single crystals are widely used in solid state lasers. These single crystals are grown by the Czochralski method. This method requires long process time and high levels of experience. Size of the crystal is limited by existence of some defects named core or facet.

Polycrystalline ceramic Nd:YAG is amore amenable product for mass production thus making it more cost efficient. Compared with YAG single crystal, transparent ceramic laser materials have the following advantages: [1]

1.easy of fabrication, 2.less expensive 3.Fabrication of large size and high concentration 4.multi-layer and multifunctional ceramic structure 5.Mass production 6.several dopants available:  $Nd^{3+}$ ,  $Er^{3+}$ ,  $Yb^{3+}$ ,  $Cr^{4+}$ ,  $Tm^{3+}$  and etc. Therefore crystal manufacturing companies are now beginning to pay attention of ceramic-laser developments.

There are two methods to fabricate of transparent polycrystalline Nd:YAG, one is conventional solid state reaction method and the other is chemical methods. Solid state reaction method requires heating at high temperatures and maybe sintering aid such as  $SiO_2$ . Consequently heat treatments at temperatures below 1600°C do not yield a single phase YAG but other phases like YAP or YAM or mixing of two phases are formed.[2-3]

By chemical methods fine powder with Nd:YAG single phase is obtained which can be sintered to transparent ceramic Nd:YAG. In order to obtain such powders, various chemical methods have been reported[4, 5, and 6]. In this study the sol-gel method was used for synthesizing of Nd:YAG

powder and it was compared with solid state reaction method.

Edita Graskite et al. reported that the single-phase Nd:YAG is formed after heating of resulted powder by sol-gel method at 1000°C [6]. In this study the monophasic Nd:YAG was not resulted by the solid state reaction method at 1350 °C but several other phases were formed. While by sol-gel method Nd:YAG single phase was synthesized at 900 °C.

## 2 Experimental Procedure

In this research the sol-gel method was applied for synthesizing of Nd:YAG powder with 4 at% Nd, and then the results were compared with solid state reaction method. In the solid state reaction, high pure  $Y_2O_3$ ,  $Nd_2O_3$  and  $Al_2O_3$  raw materials with stoichiometric amounts were used. This method consists of steps grinding, sieving, weighing, blending, pressing and sintering. The sintering step was done at the 1350 °C for 40 hr holding time. In the sol-gel method high pure  $Nd_2O_3$ ,  $Y_2O_3$  and  $Al(NO_3)_3 \cdot 9H_2O$  were used with stoichiometric amounts as the starting materials. The 4 at% Nd concentration of Nd:YAG was selected. First 27.0855 gr  $Y_2O_3$  was dissolved in acetic acid at 70-80 °C, then 1.6817 gr  $Nd_2O_3$  was dissolved in acetic acid and added to the previous solution. Also 156.239 gr Aluminum nitrate dissolved in distilled water was added. The resulting mixture was stirred for 3 hours at about 65 °C. After that 1,2-ethanediol was added to the above solution and stirred for 3 hr at 65°C. Then it was evaporated while stirring at 65 °C and finally the transparent gel was obtained. This

gel was dried at 110°C, then grounded and preheated at 800°C for 2 hr in an air atmosphere, the white powder was produced. The resulted powder was grounded again and taken two samples. One sample was heated at 900°C for 2 hr in an air atmosphere and another sample was heated at 1100°C for 9 hr in an air atmosphere.

The present phases in the heat treated powder samples of the solid state reaction and the sol-gel methods were examined by powder XRD with CuK $\alpha$  radiation (3003 pts, SEIFERT). The Study of the microstructure and morphology of the samples were performed by EDXS analysis and SEM(Model XL30, Phillips, Netherland).

### 3 Results and Discussion

The XRD patterns of two powder samples that were prepared by sol-gel method with heating to 900 °C and 1100°C at the same heating rate and heating time for 2 hr and 9 hr respectively and cooling to room temperature are shown in figures 1 and 2. These figures exhibit only the YAG phase at 900 °C and 1100°C, and they are in a good agreement with the reference of Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> (card No. 33-40). As it is observed by increasing temperature and time, peaks intensity is increased. Therefore 900°C is enough temperature to beginning crystallization into Nd:YAG and the crystallization can be completed by passing the time or increasing the temperature.

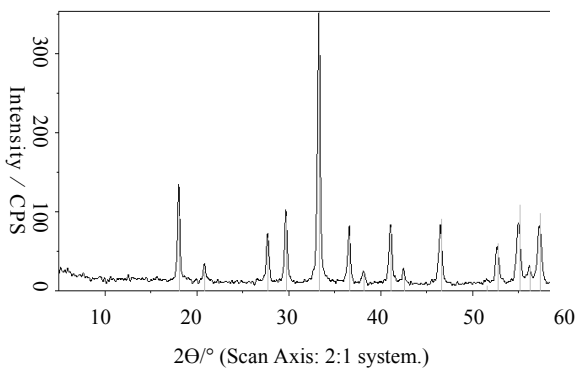


Fig.1- XRD pattern of the powder (prepared by sol-gel method) heated at 900°C for 2 hr.

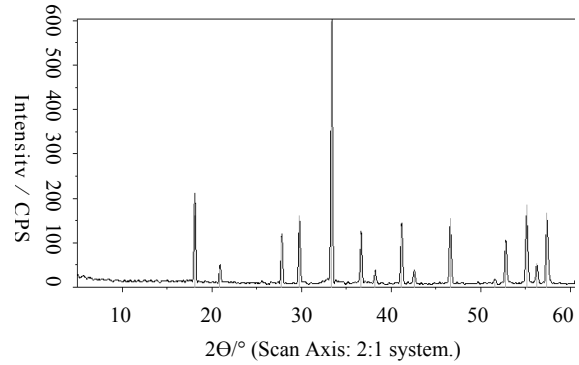


Fig.2- XRD pattern of the powder (prepared by sol-gel method) heated at 1100°C for 9 hr.

The XRD patterns of the solid state reaction method sample that was heated to 1350°C and kept for 40 hr at that temperature were shown in figure 3. As shown in this figure higher temperature and long holding time have not yielded YAG single phase, and these temperatures and times in this condition are not enough to get YAG single phase by this method, but some other phases like Al<sub>2</sub>Y<sub>4</sub>O<sub>9</sub> and Al(Nd,Y)O<sub>3</sub> were synthesized.

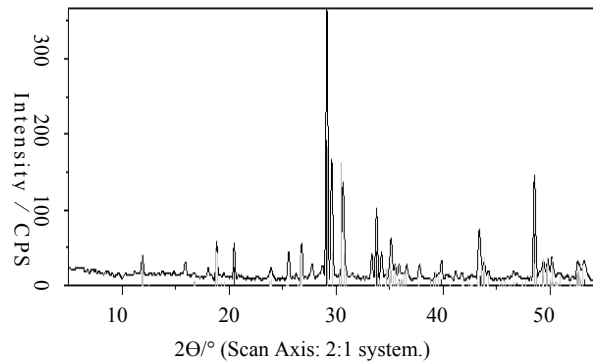


Fig.3- XRD pattern of the powder (prepared by solid state reaction method) heated at 1350°C for 40 hr

The samples prepared by sol-gel method investigated by EDAX and SEM. The distribution of Nd over the entire measuring area of the YAG phase is shown in figure 4. The bright points in this figure are Nd. This micrograph exhibits very homogeneous distribution of Nd in the structure.

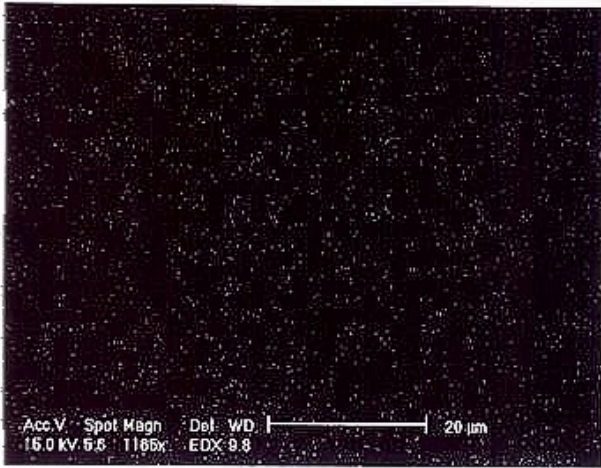


Fig.4- distribution of Nd in the YAG structure

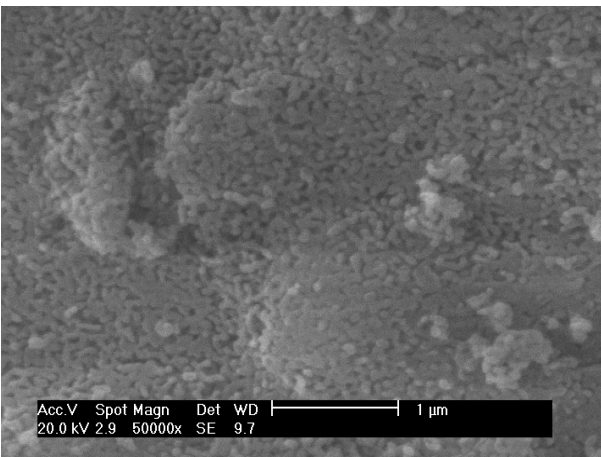


Fig.5- SEM micrograph of the powder (prepared by the sol-gel method) heated at 900 °C for 2 hr.

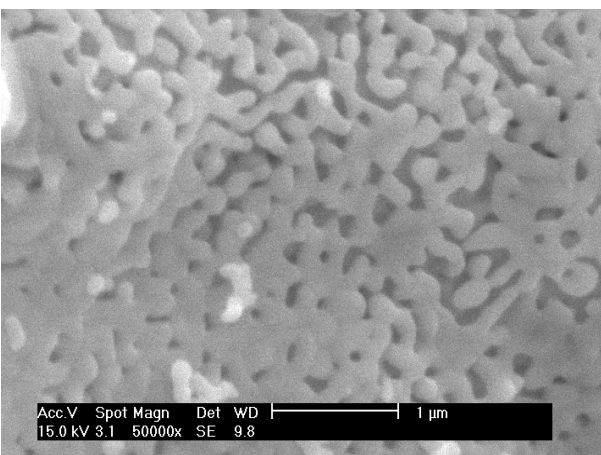


Fig.6- SEM micrograph of the powder (prepared by the sol-gel method) heated at 1100 °C for 9 hr.

The morphology of the powder samples are shown

in figures 5 and 6. By comparison of two figures, it is resulted that increasing of the heating time and temperature from 2 hr and 900 °C to 9 hr and 1100 °C cause to agglomerate powder particles and increase their sizes from ~66 nm (figure 5) to 200 nm (figure 6).

#### 4 Conclusion

Nd:YAG powder was synthesized by the sol-gel method and it was compared with solid state reaction method. The XRD analysis of prepared powder by sol-gel method showed that the YAG single phase is resulted by heat treatment at 900 °C for 2 hr and crystallization is completed by increasing time or temperature. By solid state reaction method, temperature of 1350 °C for 40 hr was not sufficient for obtaining YAG single phase. Also by sol-gel method, homogeneous distribution of Nd over the YAG phase area was obtained.

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