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Synthesis of highly doped Nd:YAG powder by SOL-GEL method

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Abstract. The sol-gel method was used for synthesizing Nd:YAG powder. The process contains several steps, for example solving in acid, mixing, evaporation etc. The XRD analysis of prepared powder by sol-gel method showed that the YAG single phase is resulted by heat treatment at 900 ºC for 2 hr. By SEM the homogeneity of Nd distribution in the YAG lattice was observed.

Keywords: laser ceramics, Nd:YAG, sol-gel synthesis.

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1. Introduction

Nd:YAG (Nd doped $Y_3Al_5O_{12}$) single crystals are widely used in solid state lasers. The method of growing these single crystals requires long process time and high level of experience and has limitation of crystal size. Therefore recently preparation of transparent ceramic Nd:YAG has been propounded. In this work sol-gel method was used for synthesizing Nd: YAG powder which can be sintered to transparent ceramic Nd:YAG for laser application [1-4].

Compared with YAG single crystal, transparent ceramic laser materials have the following advantages [5]: 1) easy of fabrication; 2) less expensive; 3) fabrication of large size and high concentration; 4) multilayer and multifunctional ceramic structure; 5) mass production 6) several dopants available: Nd^{3+},Er^{3+}, Yb^{3+}, Cr^{4+}, Tm^{3+}, *etc*.

Previously, Akin, *et al.* [6] prepared YAG powder by Self-Propagating Combustion Synthesis, then Chung *et al.* [7] used citrate gel method for synthesis of YAG powder. Also Edita Graskite *et al.* [8] reported that the single-phase Ln:YAG (Ln = Ce, Nd, Ho, Er) is formed after heating of resulted powder by sol-gel method at 1000 ºC. In the present study, single phase of Nd:YAG powder with 4 at % Nd concentration was synthesized by sol-gel method at 900 ºC.

2. Experimental procedure

High pure Nd_2O_3, Y_2O_3 and $Al(NO_3)_3.9H_2O$ 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 ºC, 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 ºC. After that 1,2-ethanediol was added to the above solution and stirred for 3 hr at 65 ºC. Then it was evaporated while stirring at 65˚ C and finally the transparent gel was obtained. This gel was dried at 110 ºC, then grounded and preheated at 800 ºC for 2 hr in an air atmosphere, the white powder was produced. The resulted powder after grinding again, was heated at 900 ºC for 2 hr in an air atmosphere.

The present phases in the heat treated powder were examined by powder XRD with CuK_α radiation (3003 pts, SEIFERT). The Study of the microstructure and morphology of the powder were performed by EDXS analysis and SEM (Model XL30, Phillips, Holland).

3. Results and discussion

The XRD pattern of the heated powder to 900 ºC for 2 hr and cooled to room temperature are shown in Fig. 1. This figure exhibit only the YAG phase, and it is in a good agreement with the reference of $Y_3Al_5O_{12}$ (card N 33-40). Therefore 900 ºC is enough temperature to resulting Nd:YAG single phase.

Also Fig. 2 shows Graskite experience that indicate the XRD pattern of an Er:YAG sample synthesized at 1100 ºC. The prepared powder investigated by EDAX and SEM. The distribution of Nd over the entire measured area of the YAG phase is shown in Fig. 3. The bright points in this figure are Nd. This micrograph exhibits very homogeneous distribution of Nd in the structure.

The morphology of the heat treated powder at 900 ºC for 2 hr, is shown in Fig. 4. In this figure necks formed between particles indicate that bondings between them have been started and therefore agglomeration is observed in some places.

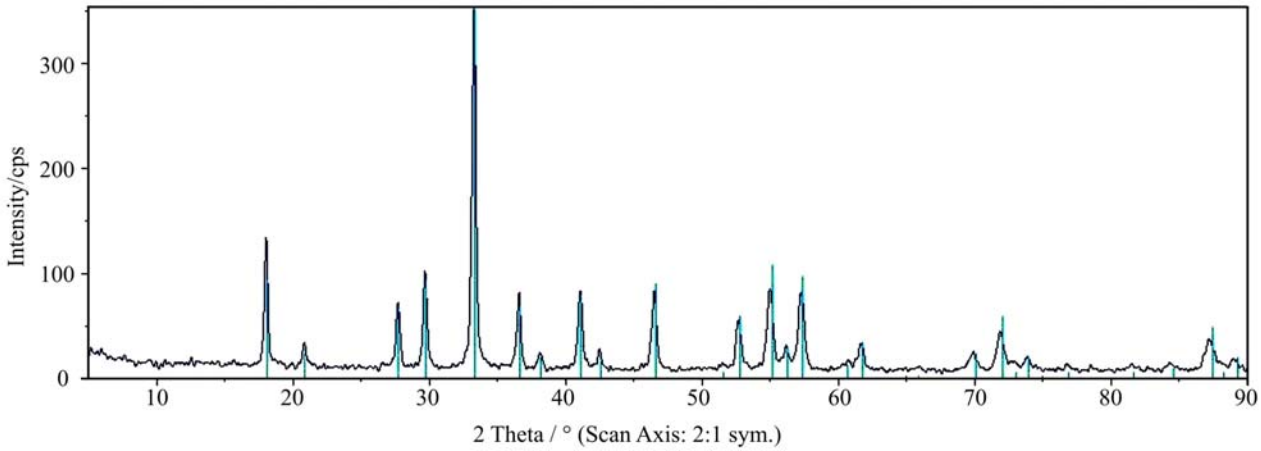

Fig. 1. XRD pattern of the powder (prepared by sol-gel method) heated at 900 ºC for 2 hr.

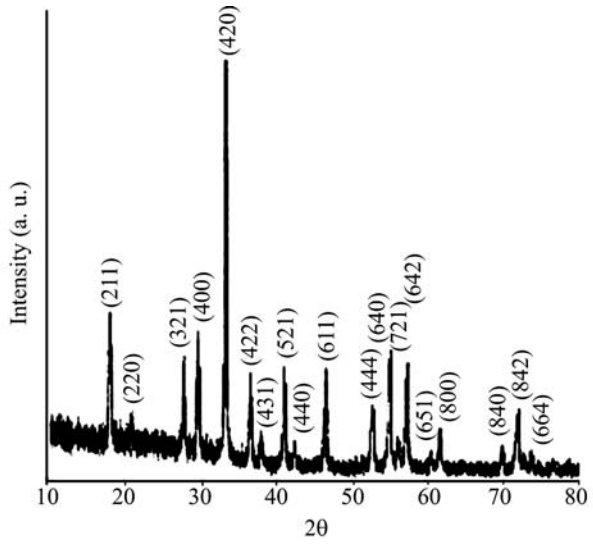

Fig. 2. XRD pattern of Er doped YAG ceramic sample synthesized by the sol-gel method at 1000 ºC (Graskite experience) [8].

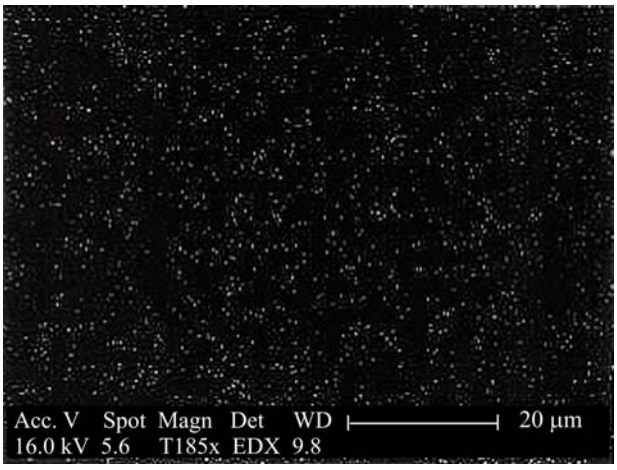

Fig. 3. Distribution of Nd in the YAG structure.

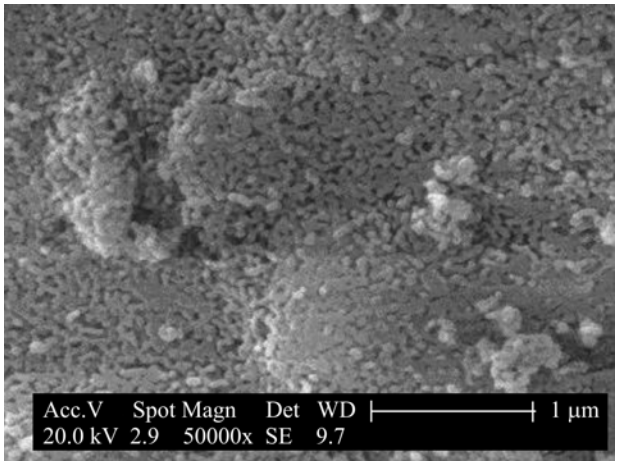

Fig. 4. SEM micrograph of the powder (prepared by the sol-gel method) heated at 900 ºC for 2 hr.

4. Conclusions

Nd:YAG powder with 4 at % Nd concentration was synthesized by the sol-gel method. The XRD analysis of prepared powder showed that the YAG single phase is resulted by heat treatment at 900 ºC for 2 hr. Also by sol-gel method, homogeneous distribution of Nd over the YAG phase area was obtained. The agglomeration of the powder particles after heating at 900 for 2 hr was observed.

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