

# Growth and lasing of single crystal YAG fibers with different Ho<sup>3+</sup> concentrations



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## ABSTRACT

A method to grow single crystal (SC) yttrium aluminum garnet (YAG) fibers with varied rare-earth ion dopant concentration has been proposed. Crystalline holmium aluminum garnet (HoAG), prepared via sol-gel process, was dip-coated on to previously grown SC YAG fibers. The HoAG coated SC YAG fiber preforms were re-grown to a smaller diameter using the laser heated pedestal growth (LHPG) technique. The final dopant concentration of the re-grown SC fiber was varied by changing the number of HoAG coatings on the preform. 120 μm diameter SC Ho:YAG fibers with four different dopant concentrations were grown. Lasing was demonstrated at 2.09 μm for these fibers. A maximum of 58.5% optical-to-optical slope efficiency was obtained.

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## 1. Introduction

Rare-earth doped yttrium aluminum garnets (Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>, YAG) have been widely used as a host material for solid-state lasers [1,2]. YAG has a broad transmission window from the UV to mid IR (about 210 nm to 5.5 μm). It has low nonlinear scattering (Stimulated Raman Scattering and Stimulated Brillouin Scattering) coefficients and better thermo-mechanical properties compared to glass laser hosts [3,4]. YAG is chemically stable and thermally durable. Its high thermal conductivity, high melting point, and low thermo-elastic coefficients make it ideal for handling high power densities. At higher operating powers, high power densities in the core limit the performance of conventional glass fibers. Under such conditions, power scaling in conventional glass fibers has limitations arising from properties of glassy silicate materials. Therefore for high power applications, rare-earth (RE) ion doped YAG single crystal (SC) fibers have been heavily studied. These systems try to utilize the superior material properties of SC YAG along with the advantages provided by a fiber geometry. Apart from providing a long interaction length and tight energy confinement, the fiber configuration

offers a large surface area to volume ratio which is advantageous for cooling.

The amount of RE ions that can be substituted for Y<sup>3+</sup> in YAG depends on the size of the substituting ion. Trivalent RE ions like ytterbium, holmium, erbium, and thulium, which are very similar in size to Y<sup>3+</sup> ions, form stable aluminum garnet phases of their own [5]. These ions can form crystalline solid solution with Y<sup>3+</sup> ions in the garnet phase at any given ratio between the two pair of ions. In contrast, RE ions which have a considerable size difference with the Y<sup>3+</sup> ions have limited solubility in YAG and have no known rare-earth aluminum garnet phase. For example, trivalent neodymium, which is about 12% larger than Y<sup>3+</sup> ion, has a maximum solubility of 18% in the YAG phase [6]. The RE dopant in the host matrix determines the output characteristics of a RE doped laser system. This is especially true for fiber lasers where the tighter confinement of the fiber geometry provides a stronger interaction of the pump and the laser wavelength with the lasing ions. For a doped fiber of a given length, there is an optimal concentration of dopant that produces maximum laser output. If the concentration is lower than this optimal value, the pump power will not be effectively absorbed by the lasing ions. If it is greater than the optimal value, the emitted laser wavelength will be reabsorbed. Hence to achieve maximum output, it is important to have a dopant concentration that is optimized for a certain length.

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Varying amounts of dopants can be introduced into a glass system with relative ease using solution doping [7]. This is due to the open amorphous nature of glasses which allow such processes to be handled at relatively lower temperatures. At these temperatures, however, YAG does not allow significant diffusion and substitution. Hence, the concentration of dopants required in a crystal fiber cannot be easily modified, and is limited to the starting material. Since SC fibers are usually grown from crystalline starting materials, trying to vary the dopant concentration would require obtaining the source material from crystalline sources which had originally been grown with the required dopant concentration. Such a process is not only time consuming but rather expensive as well. A possible solution is to use a ceramic source material, made in-house [8]. However, we have observed that the purity of the starting material has a direct influence on the optical properties of the fiber [9]. Hence, a hybrid process is desirable which allows for the variation of the dopant concentration without affecting the quality of the fiber. Such a process can not only be used to grow SC fibers with varied dopant concentration, but might be used for co-doping SC fibers with varying amounts of different RE ions. Although the present technique was developed to introduce  $\text{Ho}^{3+}$  ions into YAG, this method can be adapted for other ions like  $\text{Er}^{3+}$ ,  $\text{Tm}^{3+}$ ,  $\text{Yb}^{3+}$ , etc.

In this paper, we propose a new method to grow high purity SC YAG fibers with varied RE dopant concentrations using a low cost, low temperature sol-gel based method. A sol-gel derived layer of holmium aluminum garnet ( $\text{Ho}_3\text{Al}_5\text{O}_{12}$ , HoAG) is deposited on a  $330\text{ }\mu\text{m}$  diameter pure YAG SC fiber by dip-coating. This fiber is then re-grown to SC fibers with diameters of about  $120\text{ }\mu\text{m}$ , using the laser heated pedestal growth (LHPG) technique to introduce the  $\text{Ho}^{3+}$  ions into the YAG matrix. The dopant concentration of the  $\text{Ho}^{3+}$  ions in these fibers is changed by varying the thickness of the sol-gel derived layer. Finally, we report the lasing characteristics of these fibers at  $2.090\text{ }\mu\text{m}$  when pumped at  $1.908\text{ }\mu\text{m}$ .

## 2. Experimental

### 2.1. Sol-gel processing

Sol-gel based synthesis of rare-earth aluminum garnets has been previously studied by using an acetate-glycolate pathway by Dubnikova et al. [5]. In this study, we use a similar method to obtain polycrystalline HoAG. HoAG is derived by the hydrolysis and condensation of holmium oxide and aluminum nitrate. The materials used for the synthesis were  $\text{Ho}_2\text{O}_3$  (Alfa Aesar, 99.995%),  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  (Acros Organics, 99+%), glacial  $\text{CH}_3\text{COOH}$  (Fisher Scientific) and ethane-1,2-diol ( $(-\text{CH}_2\text{OH})_2$ ) (Acros Organics). Holmium oxide is refluxed in 0.02 M acetic acid at  $60\text{--}70\text{ }^\circ\text{C}$  for 6–8 h to obtain holmium acetate. Aluminum nitrate was added to the solution and the solution was constantly stirred at  $70\text{ }^\circ\text{C}$  for 2–3 h. Finally, ethyl glycolate is added and the final solution is kept at the same temperature, slowly evaporating the dissolved water to raise the viscosity of the precursor sol. Although the viscosity is allowed to increase considerably, gelation is not reached. The subsequent sol was used for dip coating.

The HoAG sol was applied to a SC YAG fiber with a diameter of  $335\text{ }\mu\text{m}$ , using a dip-coater illustrated in Fig. 1. Before dip-coating, the fiber is first cleaned with de-ionized water and then with isopropyl alcohol. The dip-coating was done at a withdrawal rate of  $7.5\text{ cm/min}$ . The fiber was then left to dry in air for 10 h. After this, it was put in an oven held at  $130\text{ }^\circ\text{C}$  for 60 min to dry. After drying, the fiber was sintered in air at  $1000\text{ }^\circ\text{C}$  for 14 h to remove any remaining organic material from the sol-gel precursor. This entire process was repeated for each layer. Multiple dip-coating cycles were performed to increase the thickness of the sol-gel derived

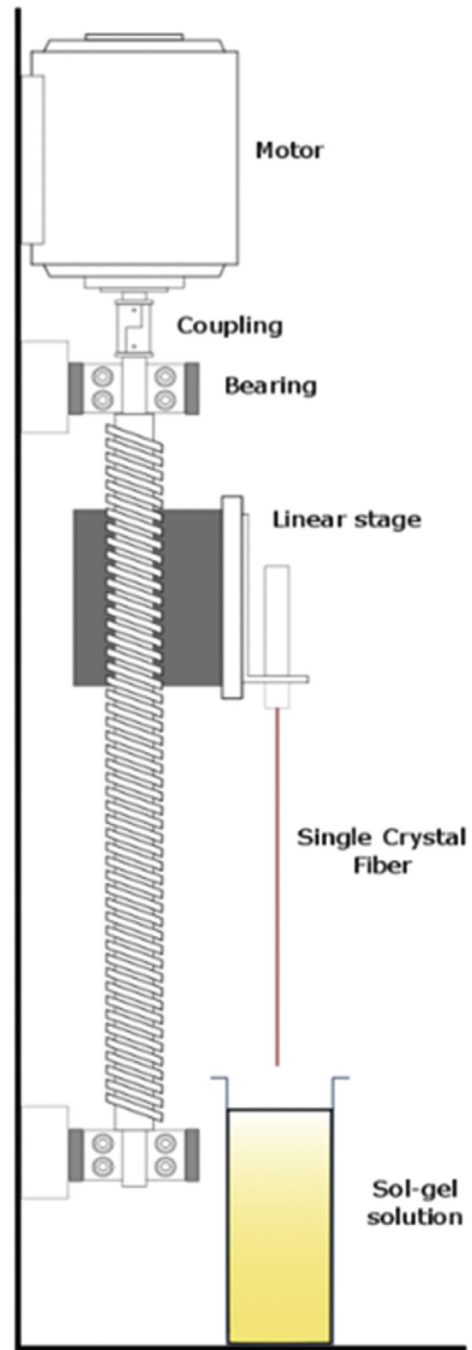


Fig. 1. Schematic of dip-coating set-up.

layer. To maintain a consistency between the layers, the same batch of sol-gel was used for all the dip-coating cycles and the withdrawal rate for the dip-coating process was kept the same. One, two, three and four cycles of dip-coating were applied on three different but contiguous sections of the YAG fiber, with each section covering roughly  $1\text{ cm}$ . The increase in thickness of the sol-gel layer due to each cycle of dip-coating was approximately  $0.25\text{ }\mu\text{m}$ .

### 2.2. Growth of SC fibers

After having the desired thickness of the sol-gel derived layer of HoAG on the pure YAG fibers, they were regrown to smaller diameter SC fibers using the LHPG technique [8,10,11]. The LHPG

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