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Resonantly pumped eye-safe Er³⁺:YAG SPS-HIP ceramic laser

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Abstract: We report for the first time laser action in resonantly-pumped transparent polycrystalline Er³⁺:YAG ceramic developed through a 2-step approach combining spark plasma sintering and HIP post treatment. Microstructural and spectroscopic properties, as well as the laser performance of large scale 0.5at.% Er:YAG transparent polycrystalline ceramic are discussed. A maximum slope efficiency of ~31% and optical-optical efficiency of 20% was measured.

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OCIS codes: (140.3380) Laser materials; (140.3500) Lasers, erbium.

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1. Introduction

Resonantly pumped solid-state eye-safe lasers are interesting sources for various applications such as remote sensing, ranging and free-space communications. Among them, $\text{Er}^{3+}:\text{YAG}$ laser emitting at $1.6\ \mu\text{m}$ [1, 2] has recently gained popularity for two main reasons: its emitting wavelength lies in a region of high atmospheric transmission and high sensor sensitivity; and resonant pumping into the upper laser manifold $^4\text{I}_{13/2}$ around $1.53\ \mu\text{m}$ ensures highly efficient operation. In addition to that, $\text{Er}^{3+}:\text{YAG}$ has been successfully used as laser medium in the Solid State Heat-Capacity Laser (SSHCL), a high-energy laser system able to provide a power output close to 5 kW [3]. Previous investigations in the last years demonstrated the scalability of the SSHCL [4]. Output powers up to $> 100\ \text{kW}$ are theoretically possible by using a single laser based on a single laser crystal. Even though the current state of the art of Czochralski growth technique is able to produce single crystal boules of several cm in diameters, core effects may

limit the useful cross section for laser experiments.

YAG transparent ceramics have attracted much attention since they can effectively replace single crystals as host materials in lasers. These polycrystalline ceramics present improved mechanical and sometimes spectroscopic properties, as well as a better heat conductivity, lower fabrication costs, larger size materials and nearly the same optical quality [5–7]. The use of ceramics is economically a highly relevant research area, furthermore, it is possible to arbitrarily introduce doping concentration profiles that mitigate thermal load inhomogeneities and thus reduce the detrimental aberrations and effects related to the thermal stresses and the thermal lensing.

Since the report in 1995 on the first polycrystalline ceramic Nd:YAG used as a gain medium, [8] numerous studies have confirmed the potential of polycrystalline ceramics in solid state lasers by reaching similar laser performances compared to single crystals [9–11].

Li *et al.* [12] reported in 2012, the successful demonstration of a 1% at. Er³⁺:YAG laser slab (10.8 mm x 4 mm x 4 mm) at 1645 nm with a slope efficiency of 51% under resonant pumping at 1532 nm. The authors also reported a maximum continuous wave laser output of 13 W. The year after, Qin *et al.* [13] published the results of an 1% at. Er:YAG ceramic slab end-pumped by an Er,Yb-fiber laser at 1532 nm with a maximum output power of 13.8 W and a slope efficiency of 54.5% at 1645 nm.

Focusing on the scale up of developed ceramics, Yagi *et al.* [11] obtained a Nd³⁺:YAG ceramic rod with a size of $\varnothing 10 \times 152$ mm by pressureless sintering presenting a laser performance comparable to that of the single crystal. Other researchers [14] reported output power using a trapezoidal geometry slab with a top face size presenting a length of 93 mm and a width of 30 mm, a bottom face size of 89 mm x 30 mm and a thickness of 3 mm processed from a Nd:YAG ceramic disk with a diameter of 130 mm and a thickness of 6 mm. The latter was developed by pressureless sintering at 1750°C for 50 h. It is worth noting that in the studies reported above, conventional sintering technique is used to produce the polycrystalline ceramics. Furthermore, in most cases, a mixture of α -Al₂O₃, Y₂O₃ and Er₂O₃ precursors prepared by ball milling, co-casting or slip-casting is employed as starting materials. Consequently, a preparation of the powder, high sintering temperatures (1700-1800°C), long holding times (10–20 h) and post-treatments are generally required to reach full density and thus, to achieve high transparency. Qin *et al.* [15] showed that fine grain boundaries (1 nm) and very low pore volume (1 ppm) are reasons of low scattering in the considered 50% at. Er³⁺:YAG ceramic materials.

Parallel to conventional ways, alternative techniques such as Pressure assisted sintering techniques, and more specifically Spark Plasma Sintering (SPS), combining the application of an external pressure during heating have been explored because it allows the consolidation of materials and the achievement of complete densification in shorter times compared to conventional processes [16, 17]. This technique was shown to be highly efficient for obtaining good optical properties in the case of the development of transparent ceramics. Transmission close to the theoretical one for different polycrystalline ceramics (yttria, alumina, spinel, etc.) developed by SPS [18–21] technique was reported in the literature. Nevertheless, only a few of them were dedicated to laser applications which require perfect optical quality and thus no defects, secondary phase or impurity. Alombert-Goget *et al.* [22] and Toci *et al.* [23] respectively performed a spectroscopic characterisation and an evaluation of laser performances of Nd³⁺-doped Lu₂O₃ transparent ceramics produced by SPS. Even if they observed laser effect, the performances were lower than the one recorded on ceramic developed by a more conventional way (HIP method). According to authors, the SPS sample exhibited higher scattering internal losses and they concluded that important efforts should be done to improve the optical quality of these samples. Sokol *et al.* [24] compared laser performances of 1 at.% Nd³⁺:YAG developed by SPS to the ones measured on the same ceramic developed by pressureless sintering. The authors reported similar optical properties such as transmission values and emission spectra between the

two ceramics but differences were found in the slope efficiency and the threshold power.

This work reports on the spectroscopic properties and laser performance evaluation of large scale 0.5at.% Er³⁺:YAG polycrystalline ceramics developed by Spark plasma sintering (SPS) and post treated with Hot Isostatic Pressing (HIP) starting from commercial powder. Therefore to improve spectroscopic properties of transparent polycrystalline ceramics, a combination of SPS and HIP processes was applied. The HIP treatment allowed to obtain highly transparent polycrystalline 0.5at.% Er³⁺:YAG ceramics. The spectroscopic results of developed ceramics were compared to the ones measured on a 0.5at.% Er:YAG single crystal grown by Czochralski method and also to the results reported in the literature for ceramics developed by pressureless sintering. To our knowledge, this is the first study reporting laser effect of a Er³⁺:YAG polycrystalline ceramic produced by the combination of non-conventional SPS technique and HIP post treatment.

2. Experimental

Commercially available high-purity 0.5at.% Er³⁺:YAG powder (Baikowski, 99.99% purity, France) whose characteristics and sintering behaviour were previously studied [25] was used as starting material. The powder had round-shaped particles with an average size of 271 nm and specific surface area of 7 m²/g. The total of impurities was less than 240 ppm including a sulphur content of 207 ppm. According to previous study, [26] 0.25 wt% of LiF was added (Sigma Aldrich, 99.995% purity) to the Er:YAG powder, then manually mixed and crushed using mortar and pestle. Finally the powder was introduced into a graphite die for SPS experiments using HP D 125 SPS facility from FCT Systeme GmbH (Rauenstein, Germany). Maximum temperature, monitored by an optical pyrometer focused on the graphite upper punch, was set to 1450°C and was maintained for 2 hours. Graphite foils were introduced both at the specimen-die and die-punches interfaces and graphite die was covered with graphite felt for thermal insulation. After sintering, the specimen was exposed to a HIP post treatment under Ar at 1400°C during 15 h and a pressure of 190 MPa in order to increase the optical quality by residual porosity removal.

SEM (NNS 450, FEI) observations were carried out on fracture surfaces of samples with a CBS (Circular BackScatter) detector and acceleration voltages of 5 kV. After mirror polishing, the in-line transmission of samples was measured using a UV-VIS-IR spectrophotometer (Cary 7000, Agilent).

To measure the emission decay time of the ⁴I_{13/2} manifold, the emission of an in house Yb/Er fiber laser emitting at 1532 nm was mechanically chopped and focused on the investigated samples. The emitted fluorescence radiation was observed perpendicularly to the exciting beam to reduce pump spurious scattering and separated from the laser pump by means of a dichroic mirror that is anti-reflection coated at 1532 nm and highly reflective (>99.9%) at 1645 nm and focused on a InGaAs detector.

3. Results and discussion

The Er³⁺:YAG transparent polycrystalline ceramic, developed following the 2-step procedure, is illustrated after polishing in Fig. 1(a). A specimen, presenting a high optical quality, with a diameter of 50 mm and a thickness of 3.8 mm was obtained. Even if high optical quality was already achieved for YAG and Er³⁺:YAG materials developed by SPS for dimension of 3 mm thickness and 30 mm diameter [25], the length required for the rod in order to evaluate material laser performances implied to proceed to a scale-up of the developed ceramics. A re-optimisation of the SPS sintering conditions and the introduction of a HIP post-treatment were necessary to keep high the optical quality (publication in progress). SEM analysis was performed on two different areas on the fracture surface as shown in Fig. 1(b) and 1(c).

SEM analysis revealed that the microstructure is not perfectly homogeneous. Some areas appear clean, without any defect (see Fig. 1(b)) while very small number of pores, exhibiting an

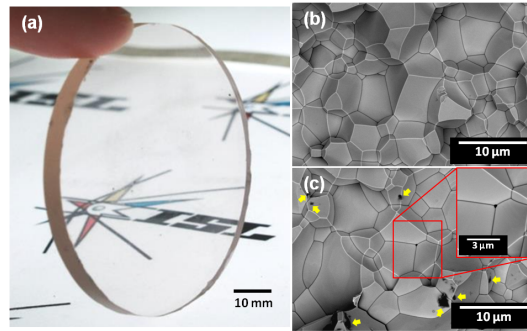


Fig. 1. (a) Er^{3+} :YAG transparent polycrystalline ceramic ($\varnothing = 50$ mm, $t = 3.8$ mm) developed by SPS and HIP post-treatment with 0.25 wt% LiF. (b) and (c) SEM images at two different areas observed of the fracture surfaces.

average size of around 300 nm, are remaining at triple points in other areas (see Fig. 1(c)). In addition, a few black spots attributed to the presence of residual carbon were observed in the same area.

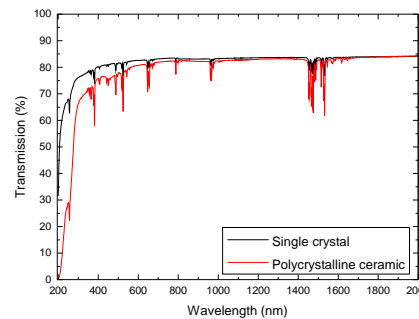


Fig. 2. In-line transmission, normalized to a thickness of 3 mm, obtained for the 0.5at.% Er^{3+} :YAG transparent polycrystalline ceramic ($\varnothing = 50$ mm, $t = 3.8$ mm) developed by SPS-HIP in the present study and the 0.5at.% Er^{3+} :YAG single crystal grown by the Czochralski method.

Figure 2 shows the transmission of the 50 mm diameter 0.5at.% Er^{3+} :YAG ceramic with a thickness of 3.8 mm processed by SPS-HIP in this study and of 0.5at.% Er^{3+} :YAG single crystal grown by using the Czochralski method. For the sake of comparison, both transmission have been normalized to a thickness of 3 mm.

The large dimensions ceramic ($\varnothing = 50$ mm, $t = 3.8$ mm) processed in this work present a T_{400} of 75.8% and T_{1100} of 82.7%. The transmission at 1100 nm is similar to the values observed in the most transparent samples reported in literature (see Table 1) and corresponds within the experimental error to the theoretical transmission where Fresnel losses only are taken into account.

In the visible range, the difference between the transmission of the investigated ceramic and the reference single crystalline sample starts increasing and becomes evident in the UV domain. These differences can be attributed to the residual porosity present in the ceramic [30–32]. Few absorbed pores at triple point with a pore diameter around 300 nm and residual carbon impurities may be responsible for transmission losses, since Mie scattering occurs when the wavelength of

Table 1. Transmission values at 400 nm and 1100 nm normalised to a thickness of 3 mm obtained for Er^{3+} :YAG transparent polycrystalline ceramics developed by SPS-HIP in the present study and those from the literature processed by pressureless sintering.

Authors	\varnothing (mm)	Thickness (mm)	T_{400} (%)	T_{1100} (%)
Zhou <i>et al.</i> [27]	20	2	76.9	84
Li <i>et al.</i> [12]	23	3.5	79.7	83
Qin <i>et al.</i> [13]	15	3	82.4	84 .1
Cavalli <i>et al.</i> [28]	10	2–3	74.3	79.8
Liu <i>et al.</i> [29]	16	4.8	79.1	8.3
Present study	50	3.8	75.8	82.7

the incident light becomes closer to the pore diameter [33]. Pores at triple points around 300 nm in size were observed in a very small number. These pores, together as residual carbon impurities, may be the responsible of the transmission losses.

Absorption spectra of the single crystal grown by the Czochralski method and the large dimensions ceramic developed by SPS-HIP were compared in Fig. 3. The ceramic sample features the same line positions and linewidths, indicating that no other crystalline phases except YAG are present.

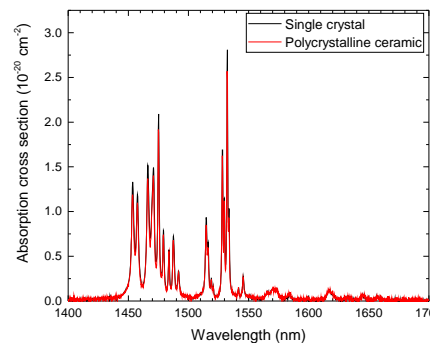


Fig. 3. Absorption spectra of the 0.5at.% Er^{3+} :YAG transparent polycrystalline ceramic ($\varnothing=50$ mm, $t=3.8$ mm) developed by SPS-HIP in the present study and the 0.5at.% Er^{3+} :YAG single crystal grown by the Czochralski method.

Lifetime values were measured for the single crystal grown by the Czochralski method and the large dimensions ceramic developed by SPS-HIP in the present work (see Fig. 4).

For all the measurements we observed the same single exponential behavior and the very similar decay time of 6.69 and 6.72 ± 0.60 ms, respectively for the single crystal and the polycrystalline ceramic, excluding in principle the presence of impurities inside the ceramics that can be sources of energy transfer mechanisms. These values are in agreement with what is reported in literature for single crystals (cf. the value of 6.9 ms reported by [34]), and are very similar to the value of 6.65 ms reported by Qin *et al.* [35] for a 0.5% at. Er:YAG polycrystalline ceramic developed by pressureless sintering.

The laser experimental set-up used is shown in Fig. 5. It consists of a single-end-pumped actively cooled 0.5% doped Er^{3+} :YAG SPS-HIP ceramic parallelepiped of 3×10 mm² cross section that is anti-reflection coated on both ends. Two samples exhibiting a length of 25 and 30 mm have been tested, this latter value being previously reported in the literature as the optimum

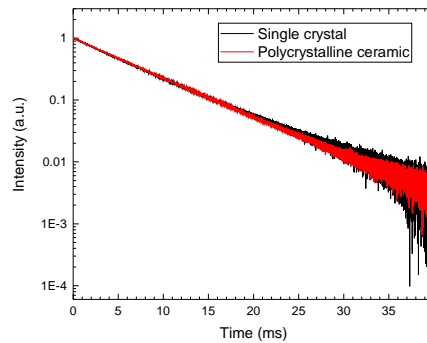


Fig. 4. Lifetime of the 0.5at.% Er^{3+} :YAG transparent polycrystalline ceramic ($\varnothing = 50$ mm, $t = 3.8$ mm) developed by SPS-HIP in the present study and the 0.5at.% Er^{3+} :YAG single crystal grown by the Czochralski method.

one for laser action in 0.5% Er^{3+} :YAG. [36] The emission of a commercial Yb,Er fiber laser, delivering up to 20 W at 1532 nm with a line width of 0.24 nm, was imaged into the gain medium by means of two lenses, providing a pump spot of ~ 300 μm diameter in the gain medium. The cavity length was chosen in order to have a fundamental mode radius of 220 μm , slightly smaller than the pump spot. This ensures a good overlap between the pump and the laser mode, allowing for optimum extracting efficiency and easy alignment.

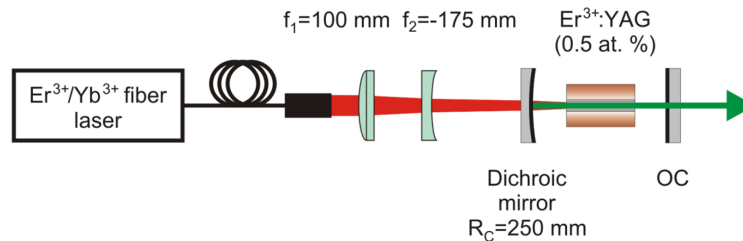


Fig. 5. Experimental set-up of the laser cavity

Figure 6(a) reports the measured slope efficiency for the sample having a length of 25 mm. For initial test, the pump laser was focused on the middle of the sample. However, we observed that the excitation of different regions inside the sample was responsible of changes in output power and slope efficiency up to a factor 2. This suggest that the optical quality in the sample is not homogeneous and that regions with higher defect concentration are present. It should be pointed out, however, that the optical quality was good enough to sustain the laser action on every chosen region. A laser threshold of 4.9 W, a maximum slope efficiency of 17.6% and an output power of 2.5 W have been obtained.

For the 30 mm we measured the output power with different output coupler (OC) reflectivity at the best pumping position, and 4.32 W of continuous wave output power was measured at 19.5 W of incident pump radiation power when using a 76% OC (see Fig. 6(b)). The maximum slope efficiency was found to be $\sim 31\%$ and optical-optical efficiency reached 20%, with a laser threshold of 4.8 W. Since the aim of this work was to evaluate the maximum power that can be extracted and due to the multimode nature of the laser resonator, an analysis of the beam quality

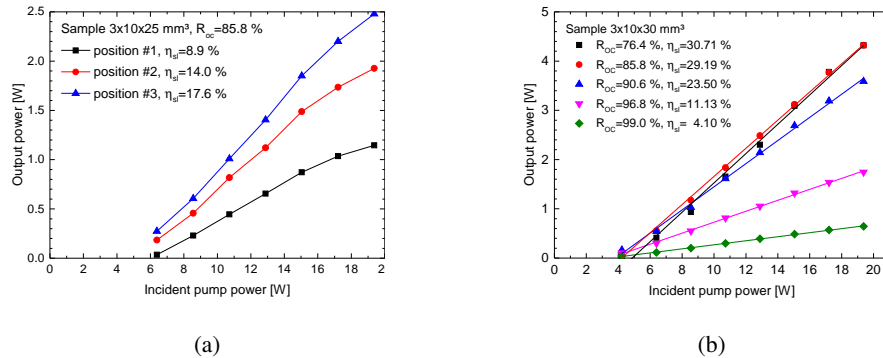


Fig. 6. CW Laser output power as a function of the incident pump power for different output coupler reflectivity.

was not performed. By evaluating the slope efficiencies η of the output power versus the pump energy for the different output coupler transmissions, a Caird-plot analysis [37] was performed to determine the cavity losses Λ and the intrinsic slope efficiency η_0 . From the fit of the curve we obtained a value of 14% for the cavity losses and 60% for the intrinsic slope efficiency. The laser performance measured are still lower than what reported in the literature for Er³⁺:YAG single crystals and conventionally sintered ceramics. This fact may be attributed to local area with higher defect concentration. By improving the homogeneity of the sintered SPS-HIP ceramics, laser efficiency comparable to the values found in the literature should be reached.

4. Conclusion

In this work, 0.5at.% Er³⁺:YAG transparent polycrystalline ceramics with a diameter of 50 mm and a thickness of 3.8 mm after polishing was elaborated by adding 0.25% wt LiF as sintering aid to the commercial powder. A 2-step approach combining SPS sintering and HIP post-treatment was used to develop a large dimension ceramic presenting a high transparency. In-line transmission values of 75.8 and 82.7% were respectively obtained at wavelengths of 400 nm and 1100 nm. These results are very close to those obtained for smaller dimensions Er³⁺:YAG ceramics developed by pressureless sintering. Compared to a 0.5at.% Er³⁺:YAG single crystal grown by the Czochralski method, transmission values are similar in the IR domain but are slightly lower in the visible range due to the presence of residual porosity. Spectroscopic properties of the large dimensions ceramic developed by SPS-HIP such as the absorption coefficient and the lifetime were close to those of the single crystal. Laser action in resonantly-pumped transparent polycrystalline Er³⁺:YAG ceramic has been observed for the first time for a SPS-HIP sintered Er:YAG transparent polycrystalline ceramic. A maximal slope efficiency of ~31% and optical-optical efficiency of 20% was measured.

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