Fabrication and Optical Property of Nd:Lu2O3 Transparent Ceramics for Solid-state Laser Applications

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Abstract: Nd3+ doped Lu2O3 crystal has been suggested to be potential gain medium for high-power solid-state lasers due to the high thermal conductivity, low phonon energy and excellent optical properties. Because of the extremely high melting point of above 2400 °C, great attention has been paid to the Lu2O3-based transparent ceramics considering the comparable optical properties and laser performance with single crystal. In this work, we aimed at fabricating highly transparent Nd:Lu2O3 ceramics and investigating the optical properties and laser performance. 1.0at%Nd:Lu2O3 ceramics were fabricated by two-step sintering, namely vacuum sintering along with hot isostatic pressing (HIP) method, from coprecipitated nano-powders. The microstructures of the as-prepared powder, green body and ceramics were studied. The average grain size of the HIPed ceramics is 724.2 nm. The final ceramic sample has the in-line transmittance of 82.4% at 1100 nm (1.0 mm thickness). The absorption cross-section of 1.0at%Nd:Lu2O3 ceramics at 806 nm is 1.50×10−20 cm2 and the calculated emission cross-section from fluorescence spectrum at 1080 nm is about 6.5×10−20 cm2. The mean fluorescence lifetime, 169 µs, of the 4F3/2→I11/2 was measured at the two excitation wavelengths of 878.8 and 895.6 nm, respectively. Laser performance of the annealed ceramic sample was investigated in quasi-continuous wave (QCW) condition. A maximum laser output power of 0.47 W with a slope efficiency of 8.7% is obtained by using an output coupler with a transmission of Toc=2.0%. Briefly, laser level Nd:Lu2O3 transparent ceramics with high optical transparency and uniform microstructure have been fabricated, which are promising gain media for solid-state lasers.

Key words: Nd:Lu2O3 transparent ceramics; co-precipitation; two-step sintering; optical property; laser performance

Considerable attention has been paid to high power solid-state lasers due to their extensive applications in many fields, such as military, industry, scientific research and medical treatments[1-3]. As an active ion for laser gain materials, the Nd3+ is widely used in high power solid-state lasers. It has a typical four-level structure, which is advantageous for reducing the pump threshold and improving the efficiency as compared to the three-level system[4-7]. Moreover, the various f-shell transitions of Nd3+ ion enable these gain media to produce abundant laser emission lines[8-9]. Generally, the electrons of Nd3+ are excited to the level of 4F5/2 after absorbing the pump light and then radiate spontaneously to the level of 4F2. Subsequently, they can jump to four possible states from 4F2, namely, 4F3/2→4I9/2, 4I11/2, 4I13/2, 4I15/2, respectively[10]. Due to the availability of several transitions, Nd3+ doped materials can achieve single-wavelength, dual-wavelength or multi-wavelength laser output by
certain design[11-15].

Yttrium aluminum garnet (YAG) is the most widely used host for high power solid-state lasers because of the excellent optical, chemical and mechanical properties[16-19]. But higher thermal conductivity favors the heat transporting which is conducive to reduce thermal effects, and allows using higher pump power densities on gain media for high power lasers[20-21]. Therefore, sesquioxides host materials such as Lu2O3[22], Sc2O3[23] and Y2O3[24] have aroused extensive attention due to the higher thermal conductivity. An attractive feature of Nd3+-doped sesquioxides gain media is that they have various emission lines in the near infrared region[10,25]. In particular, Lu2O3 appears especially promising because of the close ionic radius and mass match of Lu3+ to many lanthanide dopant ions of interest which support high doping levels with minimal sacrifice of thermal conductivity[26]. Moreover, Nd3+-doped Lu2O3 gain medium has larger emission cross section ratio between 0.9 and 1.08 μm, and higher quantum efficiency than those of Nd:YAG[8,27]. Besides, it is easy for Nd:Lu2O3 crystal to operate in the dual-wavelength regime laser. The Nd:Lu2O3 pulsed laser operation was firstly achieved with the slope efficiency of 31%[28].

However, Lu2O3 has extremely high melting point (>2490 ℃) which hampers growing high-quality single crystals. In this respect, polycrystalline transparent ceramic materials appear as promising solid-state gain media especially for materials with high melting temperatures[29-30]. In 2002, laser-level Nd:Lu2O3 transparent ceramics were firstly fabricated by vacuum sintering from nano-powders, and 10 mW laser output was obtained with the maximum absorbed power of 185 mW[31]. Afterwards, various works have shown the fabrication of Nd:Lu2O3 transparent ceramics by spark plasma sintering (SPS) even realizing the maximum laser output to 1.25 W with a slope efficiency of 38%[30,32-34]. However, using graphite mould usually causes carbon contamination of ceramics in the SPS process. Generally it is more difficult for SPS to fabricate large-size laser transparent ceramics with uniform structures compared with other techniques such as vacuum sintering or HIP. As to vacuum sintering, it’s almost impossible to remove all residual pores of transparent ceramics. While two-step sintering, namely vacuum sintering plus HIP post-treatment, can efficiently reduce the optical scattering loss of ceramics[35].

In this work, 1.0at%Nd:Lu2O3 ceramics were obtained by two-step sintering from co-precipitated nano-powder. We also investigated the microstructures, optical properties and laser performance of Nd:Lu2O3 ceramics systematically.

1 Experimental

Commercial oxide powders of Lu2O3 (99.999%, Zhongkai New Materials Co., Ltd., Jining, China) and Nd2O3 (99.99%, Alfa Aesar) were utilized as raw materials. 1.0at%Nd:Lu2O3 powder was synthesized by co-precipitation method with the optimized experimental results according to the reference [36]. The green bodies were pre-sintered at 1500 ℃ for 2 h plus HIP post-treatment at 1550 ℃ for 3 h under 150 MPa in Ar. Then final ceramics were annealed at 900 ℃ for 30 h in air, and polished to 1.0 mm thickness for further optical characterizations and laser test.

The specific surface area ($S_{BET}$) of the nano-powder was measured with the nitrogen adsorption method (BET, Quadrasorb SI, Micromeritics, USA). The phases of the precursor and powder were identified by the X-ray diffractometer (XRD, D/max2200 PC, Rigaku, Japan) with Cu Kα1 radiation (λ=0.15418 nm) at a scanning rate of 5 (°)/min in the 2θ. The morphologies of the resultant precursor and the powder, the microstructures of the thermally etched surfaces of the pre-sintered and the final ceramics were observed by the field emission scanning electron microscope (FESEM, SU8220, Hitachi, Japan). In-line transmittance and absorption spectrum of the double-sided polished ceramic, 1.0 mm of thickness, were evaluated by the UV-VIS-NIR spectrophotometer (Cary-5000, Varian, USA). The fluorescence spectrum at room temperature was performed with the fluorescence spectrometer (FLS-980, El, England).

2 Results and discussion

It can be seen from Fig. 1 that the XRD pattern of precursor includes amorphous diffraction peaks and a few obvious crystalline peaks approximately centered at an angle 2θ of 10.4°, 19.8° and 32.7°. These peaks are possibly ascribed to the carbonates, indicating that the precursor may consist of mixtures of carbonates. After calcination at 1100 ℃ for 4 h, the phase of precursor transforms into pure phase of cubic Lu2O3. The average grain size ($D_{XRD}$) of 1.0at%Nd:Lu2O3 powder is about 60.8 nm, which is calculated from the XRD spectrum by the Scherrer’s formula[37]:

$$D_{XRD} = 0.89 \lambda / (\beta \cdot \cos \theta)$$  \hspace{1cm} (1)

Where $\lambda$ is the wavelength of Cu Kα1 radiation, and $\beta$ is the full width at half-maximum (FWHM) of a diffraction peak at Bragg angle $\theta$. The theoretical density $\rho$ of the calcined powder is about 9.37 g/cm³ according to the XRD result.
The FESEM micrographs of the precursor, calcined powder and green body are shown in Fig. 2. The precursor has a 2-D loosely lamellar structure with a thickness of several tens nanometers, as it appears from Fig. 2(a). The mixtures of carbonates decompose into oxides after being calcined at 1100 °C for 4 h. It is clear that a small amount of lamellar structures still can be found and the powder consists of nanoparticles, as shown in Fig. 2(b). The average particle size ($D_{\text{BET}}$) can be obtained from the specific surface area ($S_{\text{BET}}$) of 8.254 m²/g by the formula:

$$D_{\text{BET}} = \frac{6}{\rho \cdot S_{\text{BET}}}$$  \hspace{1cm} (2)

The calculated value is 77.6 nm. The agglomeration degree of the 1.0at%Nd:Lu₂O₃ powder can be estimated by the ($D_{\text{BET}}/D_{\text{XRD}}$)³ of 2.16, indicating that the dispersity of powder still need to be improved. Firstly, the loose powder was uniaxially pressed into a disk with a diameter of 18 mm under 69 MPa. Subsequently, the green bodies were formed by cold isostatic pressing (CIP), with a relative density of 51.2%. As can be seen from Fig. 2(c), the green body is relatively compact with uniform structures, which is favorable for the fabrication of highly transparent ceramics. This also shows that dry pressing could effectively break the lamellar powder.

The green bodies were pre-sintered at 1500 °C for 2 h in vacuum without pressure. The pre-sintered ceramics are opaque after mirror-polishing. They do not possess completely dense microstructures, and their relative density measured by the Archimedes method is 96.7%.

It can be seen from the thermally etched surface of pre-sintered ceramics shown in Fig. 3(a) that there are still a large number of micro-pores located mostly at the grain boundaries. Because of the large number of pores, there is a strong light scattering in the pre-sintered ceramics to cause the opaque. However, the double-sided polished ceramics are highly transparent after HIP post-treatment at 1550 °C for 3 h under 150 MPa in Ar. Apparently, no evident micro-pores can be observed, and the thermally etched HIPed ceramic sample is nearly fully dense as shown in Fig. 3(b). The average grain size of the HIPed sample is 724.2 nm with just a little growth compared to that of the pre-sintered one (646.7 nm), which are obtained by the linear intercept method. The microstructure shown in Fig. 3(b) appears to be uniform.

The HIPed ceramics were annealed at 900 °C for 30 h and subsequently double-sided polished at laser grade with the thickness of 1.0 mm. The sample has relatively high optical quality ranging from the visible to infrared wavelength, with the in-line transmittance of 82.4% at 1100 nm and 78.3% at 400 nm, as shown in Fig. 4. The decreasing transmittance in short wavelength region may be ascribed to the tiny pores. Some residual pores of pre-sintered ceramics are removed while the rest are possibly compressed into tiny pores during the HIP process. In order to remove these residual pores in final ceramics as many as possible, the experimental parameters during the two-step sintering process are worth exploring in the following work.

Fig. 5 shows the room-temperature absorption cross section ($\sigma_{\text{abs}}$) spectrum of 1.0at%Nd:Lu₂O₃ transparent ceramics in the wavelength range from 200 to 1000 nm, which can be calculated from the absorption coefficient by:

$$\alpha = -\frac{2.303 \log(I/I_0)}{L}$$  \hspace{1cm} (3)

$$\sigma_{\text{abs}} = \frac{\alpha}{N}$$  \hspace{1cm} (4)

Where $\alpha$ is the absorption coefficient, $\log(I/I_0)$ is the optical density obtained by the spectrophotometer, $L$ is the thickness of the ceramics and $N$ is the number of the doping ions in the unit volume. The value of $N$ is
Fig. 3 FESEM micrographs of the thermally etched (1300 °C for 3 h) surfaces of 1.0at%Nd:Lu$_2$O$_3$ ceramics
(a) Ceramic pre-sintered at 1500 °C for 2 h; (b) Ceramic HIP post-treated at 1550 °C for 3 h

Fig. 4 In-line transmittance and the photograph of the double-side polished 1.0at%Nd:Lu$_2$O$_3$ transparent ceramics

Fig. 5 Room-temperature absorption cross-section spectrum of the 1.0at%Nd:Lu$_2$O$_3$ ceramics annealed at 900 °C for 30 h

cross-section could be evaluated by the Füchtbauer-Ladenburg formula:

$$\sigma(\lambda) = \frac{\lambda_{\text{peak}}^4 \beta I(\lambda_{\text{peak}})}{8\pi n^2 \tau_{\text{rad}}^\lambda} \int I(\lambda) d\lambda$$

(5)

$$n^2 = D_1 + \frac{D_2}{\lambda^2 - D_3} - D_4\lambda^2$$

(6)

Where $\lambda$ is the wavelength in μm, $\beta$ is the branching ratios of the transition of $^4F_{3/2} \rightarrow ^{11}I_{11/2}$, $I(\lambda)$ is the intensity of the room-temperature fluorescence spectrum, $n$ is the reflective index obtained by the Sellmeier equation in Ln$^3+$-Lu$_2$O$_3$ with $D_1$=3.61968, $D_2$=0.04131, $D_3$=0.00856 and $\tau_{\text{rad}}$ is the radiative lifetime of the upper laser level $^4F_{3/2}$. The theoretical value of $\tau_{\text{rad}}$ is 344 μs calculated by the Judd-Ofelt theory [10]. The calculated emission spectrum is shown in Fig. 6(b). The two strongest emission peaks of the $^4F_{3/2} \rightarrow ^{11}I_{11/2}$ transition locate at 1076 and 1080 nm with the maximum emission cross-section at 1080 nm of about $6.5 \times 10^{-20} \text{cm}^2$.

The measurement of the upper level lifetime of 1.0at%Nd:Lu$_2$O$_3$ ceramics was carried out with the excitation radiation tuned either to 878.8 nm or 895.6 nm, respectively. These wavelengths correspond to the absorption of the $^4I_{9/2} \rightarrow ^4F_{3/2}$ transition of electrons for Nd$^{3+}$ in sites with C$_2$ symmetry, which is the most
important site for laser emission as the Nd$^{3+}$ ions in the sites with C$_{3i}$ symmetry have a small emission and absorption cross section$^{[33]}$. The obtained fluorescence decay behaviors with excitation at the two different wavelengths are substantially identical, as it can be seen from the results shown in Fig. 7. The mean fluorescence lifetime can also be evaluated numerically with the following formula:

$$\tau_{\text{mean}} = \frac{1}{I(t = 0)} \int_{0}^{\infty} I(t) \, dt$$

(7)

Where \( t \) is the excitation time. Using the Eq. (7), when the ceramic sample is respectively excited at 878.8 and 895.6 nm, we obtain the same value of the lifetime, \( \tau_{\text{mean}}=169 \mu s \). This value is relatively higher than the value of 153 \( \mu s \) measured for 1.0at\%Nd:Lu$_2$O$_3$ ceramics from the reference $^{[34]}$, but substantially lower than the theoretical value of 344 \( \mu s \) calculated from J-O theory. It is also lower than the value of the single crystal$^{[10]}$. This reduced lifetime may be addressed to the lattice defects and concentration quenching effect.

The laser test was performed using the laser cavity layout shown in Fig. 8, similar to that for Yb-doped materials$^{[39]}$. The sample was soldered by a sheet of indium on a copper heat-sink and cooled by water at 17 \( ^\circ C \), and pumped by a fiber-coupled LD. It emits at 789.3 nm (FWHM 2.4 nm) with an almost cylindrical intensity distribution in the region of the focal plane (i.e. beam radius 50 \( \mu m \), measured with a CCD camera), and numerical aperture of the pump beam is 0.22. The resonator cavity consists of various optical elements such as End-Mirror (EM), Folding-Mirror (FM, spherical, ROC=150 mm) and Flat Output Coupler mirror (OC). Both EM and FM are dichroic (high transmission for the pump wavelength, high reflection between 980 and 1100 nm) and 80 mm apart. The distance between FM and OC is 137 mm. The output power is measured by the power meter M1, and a filter is used to reject the residual pump radiation, preventing it from reaching the power meter. The transmission of the filter at the lasing wavelength, i.e. 81.5%, was considered in the evaluation of the actual output power values.

The 1.0at\%Nd:Lu$_2$O$_3$ ceramic sample was pumped in QCW mode (10 Hz of repetition rate, duty factor of DF=20\%) in order to limit the thermal load into the ceramic sample. The maximum absorbed pump power by the sample was 5.9 W, corresponding to 33 W of incident pump power. Under lasing conditions, 18\% of the incident pump power was absorbed by the sample. Laser output was successively extracted through OCs with different transmission values (\( T_{\text{OC}}=2.0\%, 5.2\%, 7.0\%, 19.0\% \)) to find the optimal output coupling transmission. The lasing wavelength was measured by a fiber-coupled spectrometer with a resolution of 0.4 nm along the entire wavelengths range.

Fig. 9(a) shows the output power as a function of the absorbed pump power for the 1.0at\%Nd:Lu$_2$O$_3$ ceramic sample under test using output couplers with various transmission values. All main laser emission parameters are reported in Table 1. It can be seen that the laser threshold increases by increasing the transmission value of OC mirror. The slope efficiencies are similar for the OCs with transmission values ranging from 2.0\% to 7.0\%, whereas the maximum optical efficiency, that is 7.8\%, was obtained by $T_{\text{OC}}=2.0\%$. The slope efficiency and laser output power are higher than those of the Nd:Lu$_2$O$_3$ ceramics obtained by vacuum sintering$^{[31]}$. However, the laser performance needs to be improved by further decreasing the optical loss of Nd:Lu$_2$O$_3$ ceramics.

Fig. 9(b) reports the two highest emission peaks acquire with 4.8 W of absorbed pump power when the cavity is closed with different OCs. The laser emits simultaneously on two different wavelengths respectively at 1076 nm and at 1080.3 nm, corresponding to the transition from the level \( ^{4}F_{3/2} \) to the \( ^{4}I_{11/2} \). It appears that the main emission peaks at about 1076 nm, with a fraction (between 20\% and 30\%) of emission peaking at 1080 nm.
1.50

The Nd³⁺ ion and the absorption cross-section at 806 nm was due to the large number of f-shell transitions of Nd³⁺ ions concentration. The ceramic sample was pumped in QCW with a maximum laser output power of 0.47 W corresponding to a slope efficiency of 8.7% (T₀=2.0%). The tested laser performance demonstrated that the optical quality of Nd:Lu₂O₃ ceramics still remained to be improved, such as the microstructures, the optical uniformity and the surface quality of Nd:Lu₂O₃ ceramics, to reduce the optical loss.

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### Table 1 Main laser emission parameters

| Output coupler transmission/ % | Maximum power/ W | Slope efficiency/ % | Optical efficiency/ %* | Laser threshold/ W |
|-----------------------------|------------------|---------------------|------------------------|-------------------|
| 2.0                         | 0.460            | 8.7                 | 7.8                    | 0.57              |
| 5.2                         | 0.372            | 8.9                 | 6.3                    | 0.81              |
| 7.0                         | 0.325            | 8.3                 | 5.7                    | 1.20              |
| 19.0                        | 0.029            | 1.9                 | 4.4                    | 4.20              |

*: Calculated with respect to the absorbed pump power

### 3 Conclusion

1.0at%Nd:Lu₂O₃ transparent ceramics were fabricated by vacuum sintering plus HIP methods from the co-precipitated nano-powder in this work. The microstructures of powder, green body and ceramics were studied in detail. The green bodies showed a relatively compact and uniform structure from the lamellar nano-powder. Porous ceramics pre-sintered in vacuum for short time finally turned into completely dense ceramics after HIP post-treatment. The in-line transmittance of 1.0at%Nd:Lu₂O₃ ceramics with the thickness of 1.0 mm was 82.4% at 1100 nm after annealing.

We have also systematically investigated the spectral properties and the laser performance of 1.0at%Nd:Lu₂O₃ ceramics. The ceramic sample showed many absorption bands due to the large number of f-shell transitions of Nd³⁺ ion and the absorption cross-section at 806 nm was 1.50×10⁻²⁰ cm². The strongest two emission peaks of the ⁴F₃/₂→⁴I₁₁/₂ transition locating at 1076 and 1080 nm with the maximum emission cross-section at 1080 nm of about 6.5×10⁻²⁰ cm², which was calculated from the fluorescence spectrum. The fluorescence lifetime of the upper level ⁴F₃/₂ measured at the two excitation wavelengths of 878.8 or 895.6 nm was 169 μs, which is lower than the theoretical value. This was possibly ascribed to the lattice defects of Lu₂O₃ ceramics and the quenching of Nd³⁺ ions concentration. The ceramic sample was pumped in QCW with a maximum laser output power of 0.47 W corresponding to a slope efficiency of 8.7% (T₀=2.0%). The tested laser performance demonstrated that the optical quality of Nd:Lu₂O₃ ceramics still remained to be improved, such as the microstructures, the optical uniformity and the surface quality of Nd:Lu₂O₃ ceramics, to reduce the optical loss.

![Fig. 9 Laser output power vs incident pump power (a) and emission spectra for different transmission values of the OC (b)](image-url)
固体激光用 Nd:Lu₂O₃ 透明陶瓷的制备和光学性能研究

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摘 要: Nd:Lu₂O₃ 材料由于具有高热导率、低声子能量和优异的光学特性而成为非常有前景的高功率固体激光器用的增益介质。但 Lu₂O₃ 单晶的熔点超过 2400 ℃，难以生长，而 Lu₂O₃ 陶瓷既能在低温下制备，又具有与晶体相当的光学性质和激光性能从而备受关注。本研究制备了高透明的 Nd:Lu₂O₃ 陶瓷并对其光学性质和激光性能进行探究。以共沉淀法制备的纳米颗粒为原料，采用真空烧结结合热等静压 (HIP) 两步烧结法制备了 1.0at%Nd:Lu₂O₃ 透明陶瓷。对制备的粉体、素坯和陶瓷的微结构进行了表征：HIP 后处理的陶瓷平均晶粒尺寸为 724.2 nm。厚度为 1.0 mm 的 1.0at%Nd:Lu₂O₃ 透明陶瓷在 1100 nm 处的透射率通过测量是 82.4%，样品在 806 nm 处的吸收截面为 1.50×10⁻²⁸ cm²，而根据荧光光谱计算得到的发射截面为 6.5×10⁻²⁶ cm²。在 878.8 和 895.6 nm 波长激发下，1.0at%Nd:Lu₂O₃ 透明陶瓷在 1000 nm 处的荧光强度为 169 μV。当输出耦合器的透射率 TED=2.0%时，退火后的 1.0at%Nd:Lu₂O₃ 透明陶瓷获得了最大输出功率为 0.47 W 的准连续 (QCW) 荧光输出，斜率效率为 8.7%。本研究成功制备了显示均匀、高透明度的 1.0at%Nd:Lu₂O₃ 陶瓷，并展示了其在固体激光增益介质领域的广阔应用潜力。

关 键 词: Nd:Lu₂O₃ 透明陶瓷; 共沉淀法; 两步烧结; 光学性能; 激光性能