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High performance of La-doped Y$_2$O$_3$ transparent ceramics

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Abstract: As an optical material, Y$_2$O$_3$ transparent ceramics are desirable for application as laser host materials. However, it is difficult to sinter and dense of Y$_2$O$_3$ hinders the preparation of high-quality optical ceramics via traditional processes. In this work, we use La$_2$O$_3$ as a sintering aid for fabricating high-transparency Y$_2$O$_3$ ceramics using a vacuum sintering process. It is demonstrated that the in-line optical transmittance of 15.0 at% La-doped Y$_2$O$_3$ at a wavelength of 1100 nm achieves a transmittance of 81.2%. A sintering kinetics analysis reveals that a grain-boundary-diffusion-controlled mechanism dominates the faster densification at high La$^{3+}$ concentrations. It is also shown that both the mechanical and thermal properties of Y$_2$O$_3$ transparent ceramics are significantly improved upon the increase of La$_2$O$_3$ sintering additives. The results indicate that a La-doped Y$_2$O$_3$ transparent ceramic is a promising candidate for a laser host material.

Keywords: Y$_2$O$_3$ transparent ceramics; optical transmittance; sintering kinetics; mechanical properties; thermal properties

1 Introduction

As an attractive optical material, Y$_3$O$_5$ has attracted significant interest for use in infrared dome, refractory, and semi-conductor devices and up-converters, owing to its broad transmission region, high refractive index, high corrosion resistance, low phonon energy, etc. [1]. In that regard, a typical cubic crystal of Y$_3$O$_5$ is suitable for laser ceramics. Compared to Y$_3$Al$_5$O$_{12}$ (thermal conductivity of 10.5 W/(m·K) and thermal expansion coefficient of 7.8×10$^{-6}$ K$^{-1}$), Y$_2$O$_3$ has superior thermal properties with higher thermal conductivity of 13.6 W/(m·K) and lower thermal expansion coefficient of 6×10$^{-6}$ K$^{-1}$, which are beneficial for minimizing the thermal lensing effect and improving the laser beam quality, and thus is attracting more attention for use as a high-output-power laser host material [2–7].

Since the first demonstration of using YAG-based transparent ceramics for lasers in 1995 [8], the development of transparent ceramics has offered potential advantages over single crystals for metal rare-earth sesquioxide Y$_2$O$_3$ materials [9,10]. It is known that the heat energy generated from operating a
high-power laser will congregate and induce thermo-optic aberrations, lensing, and birefringence, deteriorating the operation of laser host medium [11–13]. In addition, the mechanical properties, representing the interatomic and inter-particle strengths, are very important in mechanical industrial applications of laser materials [14]. Therefore, there is an urgent need to produce transparent Y₂O₃ ceramics with excellent thermal and mechanical properties.

A vacuum sintering procedure is considered to be an efficient technique for fabricating highly transparent ceramics, with technological and economic advantages [15–17]. In addition, a sintering additive is beneficial for lowering the sintering temperature and shortening the sintering time during the vacuum sintering process [18–23]. However, it has been found that sintering aids have a significant effect on the mechanical and thermal properties (in addition to the physical characteristics), probably owing to a mismatch of ion radius and mass [24,25].

Anti-sintering properties of Y₂O₃ have hindered the use of the conventional growth method for preparation of a dense sample with good optical quality. In this work, La₂O₃ is added as sintering aid, and high transparency in Y₂O₃ laser host materials is obtained by using a vacuum sintering process. Sintering kinetics is applied to analyse the densification and grain-growth mechanisms of different sintering additive concentrations. Furthermore, the effects of the sintering additives on the mechanical and thermal properties are also examined in detail.

2 Experimental procedure

2.1 Sample fabrication

High-purity commercial Y₂O₃ (99.99%, Shanghai Yuelong New Materials Co. Ltd., Shanghai, China) is used as a raw material, and La₂O₃ (99.99%, First Rare Materials Co. Ltd., Qingyuan, China) powder is adopted as the sintering additive. As the structures of Y₂O₃ (cubic) and La₂O₃ (hexagonal) are different, the solid solubility of the La-doping concentration is within 15.0 at% [26,27]. Therefore, the samples are designed for 5.0, 10.0, and 15.0 at% La-doping content. The details of the sample fabrication are similar to the processing in our previous research [28].

2.2 Characterization

The optical transmittances of the specimens are identified by an UV/VIS/NIR spectrometer (UV-3600, Shimadzu, Tokyo, Japan) at wavelengths of 300–2000 nm, with the spectral resolution 1–2 nm. The phase compositions of samples are determined via X-ray diffraction (XRD, D/max-2500, Cu Kα radiation, Rigaku Smart Lab, Japan). The morphologies are characterised using field-emission scanning electron microscopy (Zeiss Merlin, Germany), whereas the diameters of the particles are analysed using the obtained SEM images and ImageJ software. The bulk density of a sample is measured using the Archimedes method. To achieve the sound wave velocities of the samples, both v_l and v_t are determined by ultrasonic method using an Ultrasonic Pulser/Received machine (5900 PR, Panametrics-NDT, Waltham, USA), and the following equation is used [29]:

\[ v_i = \frac{2d}{t_i} \]  

Here, \( i = l \) and \( t \) denote the longitudinal and transverse modes, respectively, \( d \) is the thickness of the sample (2 mm), and \( t_i \) is measured between two successive echoes for the longitudinal and transverse modes. The mechanical parameters (shear modulus (\( G \)), bulk modulus (\( B \)), Young’s modulus (\( E \)), Pugh’s modulus ratio (\( k \)), Poisson’s ratio (\( \sigma \)), and Vickers hardness (\( H_v \)) are estimated from Eqs. (2)–(6) [30–32]:

\[ G = \rho v_i^2 \]  
\[ B = \rho \left( v_i^2 - \frac{4}{3} v_t^2 \right) \]  
\[ E = \frac{9BG}{3B+G} \]  
\[ k = \frac{G}{B} \]  
\[ \sigma = \frac{1-2\left(\frac{v_t}{v_i}\right)^2}{2-2\left(\frac{v_t}{v_i}\right)^2} \]  
\[ H_v = 2.0(k^2G)^{0.585} - 3.0 \]

The measurement of the thermal properties can be obtained in our previous work [33]. The thermal diffusivity coefficient (\( \lambda \)) of sample (\( \phi 10.0 \text{ mm} \times 1.0 \text{ mm} \)) is measured using the laser flash method by a laser flash apparatus (LFA427, Netzsch, Selb, Germany). The heat capacity (\( C_p \)) is determined by differential
scanning calorimeter using a sapphire standard (DSC403PC, Netzsch, Selb, Germany). The relative densities ($\rho$) are obtained by dimensional and mass determinations on cylindrical slab sample ($\phi$ 13.0 mm × 2.0 mm).

3 Results and discussion

3.1 Microstructure and phase

The optical transmittance is known as the key performance indicator in the application of transparent ceramics. As shown in Fig. 1(a), after 1750 °C sintering for 15 h, the in-line transmittances increase significantly at a wavelength of 400–2000 nm as the La doing increases, and the maximum value of the in-line transmittances achieves 80.2%. Evidently, a significant enhancement of optical transmittance is obtained owing to the significantly increased La$_3^+$ concentration. Figure 1(b) illustrates the XRD patterns of the La-doped Y$_2$O$_3$ ceramics. As the concentration of La-doping increases, each peak matches well with the Y$_2$O$_3$ cubic phase (JCPDS 41-1105), and the diffraction peaks move to lower angles, implying the increasing of lattice constant. The changes of lattice parameters can be attributed to the difference of radius between La$_3^+$ ion (1.06 Å) and Y$_3^+$ ion (0.89 Å) [34]. As shown in the inset picture of Fig. 1(b), unit-cell parameters increase with La$_3^+$ concentration increasing. Furthermore, no second phase is detected, implying that La$_3^+$ is completely soluble in the matrix material.

As shown in Fig. 2, the average grain size increases with a decrease in La concentration for the La-doped Y$_2$O$_3$ ceramic at a sintering temperature of 1750 °C for 15 h. For example, the 5.0 at% La-doped sample has an average grain size of approximately 35 μm and local compositional inhomogeneity, as compared to the 15.0 at% La-doped Y$_2$O$_3$, which has an average grain size of approximately 18 μm and a pore-free or heterogeneous phase. This implies that the grain size and pores could be remarkably suppressed by an increase of La$_3^+$ concentration [35,36].

3.2 Sintering kinetics analysis

As shown in Fig. 3(a), the effects of both the temperature and La$_3^+$ doping concentration on the transparency of the samples are evident. One can see that, at a fixed doping concentration, the optical transmission increases with increasing temperature for the samples, in agreement with the findings of Zhou et al. [19]. Moreover, the transparency increases significantly with an increase of sintering additive content at a certain temperature, which confirmed by the in-line transmittance results.

To probe the densification mechanisms of La-doped Y$_2$O$_3$ transparent ceramics, we conducted a kinetics analysis of samples with varied sintering processing. Figure 3(b) plots the relative density vs. sintering temperature for the 5.0, 10.0, and 15.0 at% La-doped Y$_2$O$_3$ ceramics. Sintering time kept 10 h at each sintering temperature. It is shown that, for both the 5.0 and 10.0 at% La-doped, the sample densification occurred from 1600 to 1800 °C. In contrast, no increase in the relative density appeared as the sintering temperature increased to higher than 1700 °C for the 15.0 at% La-doped Y$_2$O$_3$ ceramics. Thus, a high sintering additive content can significantly reduce the sintering temperature. In addition, the densification parameters increase with La$_3^+$ concentration increasing. Furthermore, no second phase is detected, implying that La$_3^+$ is completely soluble in the matrix material.

As shown in Fig. 2, the average grain size increases with a decrease in La concentration for the La-doped Y$_2$O$_3$ ceramic at a sintering temperature of 1750 °C for 15 h. For example, the 5.0 at% La-doped sample has an average grain size of approximately 35 μm and local compositional inhomogeneity, as compared to the 15.0 at% La-doped Y$_2$O$_3$, which has an average grain size of approximately 18 μm and a pore-free or heterogeneous phase. This implies that the grain size and pores could be remarkably suppressed by an increase of La$_3^+$ concentration [35,36].

![Fig. 1](a) In-line transmittance and (b) X-ray diffraction (XRD) patterns of 5.0, 10.0, and 15.0 at% La-doped Y$_2$O$_3$ ceramics under a sintering temperature of 1750 °C for 15 h.
rate can be calculated by [37]:

\[
\frac{d\rho}{dt} = \frac{C \gamma_s DN_g}{G^n}
\]  \hspace{1cm} (8)

In the above, \(\rho\) is the density of the sample, \(C\) is a constant, \(\gamma_s\) is the surface energy for solid/gas, \(D\) is either the coefficient of the lattice diffusion (\(D_L\)) or the coefficient of the grain-boundary diffusion (\(D_b\)), \(N_g\) is the number of pores per grain (assuming \(N_g\) is a constant here), and \(G\) is the average grain size. In that regard, \(n\) is a constant on behalf of the grain-size exponent. Here, the densification mechanism is dependent on the value of the constant \(n\). It is known that a lattice-diffusion control mechanism is confirmed when \(n = 3\), whereas a grain-boundary-diffusion control mechanism is dominant when \(n = 4\). As shown in Figs. 3(c) and 3(d), according to Eq. (8), the \(n\) value of the samples is fit to 3.6, 3.8, and 4.2, corresponding to the 5.0, 10.0 and 15.0 at% La-doped samples, respectively. This indicates that the grain-boundary-diffusion control mechanism is the predominant densification process for La-doped \(\text{Y}_2\text{O}_3\). The densification rates of the samples with 5.0, 10.0, and 15.0 at% La-doped concentrations are 9, 12, and 18 [38], respectively. Thus, the \(\text{Y}_2\text{O}_3\) ceramic with 15.0 at% La-doping shows the highest rate of grain-boundary diffusion. This is possibly due to the difference in ionic radius between the La\(^{3+}\) ion and Y\(^{3+}\) ion, which is beneficial for generating elastic strain energy and promoting the diffusion of the Y\(^{3+}\) ion in \(\text{Y}_2\text{O}_3\) [39,40].

Additionally, the grain-growth kinetics can be calculated, for further study of the sintering mechanisms. A grain-growth rate equation is applied for the \(\text{Y}_2\text{O}_3\) ceramics as follows [34]:

\[
G_t^n - G_0^n = k(t - t_0)
\]  \hspace{1cm} (9)

Here, \(G_0\) and \(G_t\) are the grain sizes with sintering time
of $t_0$ and $t$, respectively, $m$ is the exponent of the grain growth, and $k$ is the rate constant (confirmed using data of grain growth). From Figs. 4(a) and 4(b), the experimental values of $m$ are fit to a line with a slope of approximately 3, and the $k$ values of the samples with 5.0, 10.0, and 15.0 at% La are $1.3 \times 10^{-16}$, $7.9 \times 10^{-17}$, and $2.1 \times 10^{-17}$ m$^3$/s, respectively. This indicates that higher La$^{3+}$ doping results in inhibiting the grain-growth process.

The activation energies of the samples sintered from 1600 to 1800 °C for 2–10 h are calculated according to the Arrhenius equation [41]:

$$\ln k = \frac{-E_a}{RT} + C \tag{10}$$

In the above, $k$ is the rate constant from Eq. (9), $E_a$ is the activation energy, and both $R$ and $C$ are constants. As shown in Fig. 4(c), we fit $\ln k$, and obtain the activation energies $E_a = 710$, 670, and 650 kJ/mol for the samples with 5.0, 10.0, and 15.0 at% La concentrations, respectively, which corresponds to the findings of Borovkova et al. [42]. According to a previous kinetic analysis, the grain-size-density ($G$–$\rho$) trajectory of La-doped $Y_2O_3$ with a sintering temperature of 1750 °C for 2–40 h is presented in Fig. 4(d). It is found that the increase of La concentration promotes the densification rate, but reduces the grain-growth rate. Thus, the faster densification kinetics in $Y_2O_3$ samples with a higher La concentration leads to transparency at a lower sintering temperature, as confirmed through the optical experimental results.

### 3.3 Mechanical properties

Generally speaking, mechanical properties are very important for the application of laser matrix materials [43]. In this work, the mechanical parameters ($B$, $G$, $E$, $k$, $\sigma$, and $H_v$) are obtained by calculation from the longitudinal and transverse sound velocities. As shown in Table 1, the observed values of $G$ are much smaller than those of $B$, indicating that $G$ is a key parameter for the mechanical stability of La-doped samples. In particular, the values of $G$ in $Y_2O_3$ doped with 5.0 at% La are higher than those in $Y_2O_3$ ceramics, owing to
the smaller particle size [50]. It is well known that $k$ ($G/B$) refers to the brittle and ductile behaviour of materials, and the critical value for distinguishing ductility from brittleness is approximately 0.57. From Table 1, the $k$ values are 0.50, 0.47, and 0.46 for the 5.0, 10.0, and 15.0 at% La$^{3+}$ concentrations, respectively. In particular, the $k$ value of the 15.0 at% La-doped Y$_2$O$_3$ is approximately 0.46, corresponding to a brittle material. Furthermore, the values of $\sigma$ (as determined from $G$ and $E$ data) are 0.285, 0.298, and 0.303 for the 5.0, 10.0, and 15.0 at% La-doped Y$_2$O$_3$, respectively, consistent with the reported values of Ramzan et al. [45]. Moreover, the values of $H_v$ for the La-doped Y$_2$O$_3$ increase with increasing La concentrations owing to the Hall–Petch effect [47], and are still higher than the values of a Y$_2$O$_3$ single crystal [44]. In addition, from Table 1, it can be observed that the value of $E$ of the 15.0 at% La-doped Y$_2$O$_3$ is approximately 179.8, which is beneficial to alleviating stress accumulations in the host material.

![Figure 4](image.png)

**Fig. 4** (a) Grain size and (b) grain growth kinetics as a function of the sintering time for La-doped Y$_2$O$_3$ ceramics at a sintering temperature of 1750 °C. (c) Arrhenius analysis of samples at a sintering temperature of 1650–1800 °C for 2–10 h. (d) Grain size vs. relative density of samples at a sintering temperature of 1750 °C for 2–40 h.

**Table 1** Mechanical properties and sound wave velocities of La-doped Y$_2$O$_3$ ceramics

| La concentration (at%) | $v_l$ (m/s) | $v_t$ (m/s) | $B$ (GPa) | $G$ (GPa) | $k$ | $E$ (GPa) | $\sigma$ | $H_v$ (GPa) |
|------------------------|-------------|-------------|-----------|-----------|-----|-----------|---------|-------------|
| 5.0                    | 6802        | 3733        | 139.1     | 70.0      | 0.50| 172.0     | 0.285   | 7.1         |
| 10.0                   | 6786        | 3637        | 144.5     | 67.3      | 0.47| 174.8     | 0.298   | 7.8         |
| 15.0                   | 6733        | 3582        | 145.3     | 66.0      | 0.46| 179.8     | 0.303   | 8.2         |
| Y$_2$O$_3$ crystal [44]| —           | —           | 149.5     | 66.3      | 0.44| 173.0     | 0.307   | 7.6         |
| Y$_2$O$_3$ crystal [45]| —           | —           | 153.8     | 62.4      | 0.41| 165.0     | 0.320   | —           |
| Y$_2$O$_3$ ceramic [46]| 6931        | 3712        | 148.9     | 69.2      | 0.46| 179.8     | 0.299   | —           |
| Y$_2$O$_3$ ceramic [47]| 6876        | 3670        | —         | —         | —   | —         | —       | —           |
| La–Y$_2$O$_3$ ceramic [48]| 6680        | 3640        | 142.0     | 65.0      | 0.46| 170.0     | 0.300   | 8.8         |
| La–Y$_2$O$_3$ ceramic [49]| 6687        | 3522        | 144.5     | 63.6      | 0.44| 144.5     | 0.308   | —           |
3.4 Thermal properties

In laser transparent ceramics, the thermal expansion coefficient is closely related to the material life. To obtain the thermal expansion coefficient ($\alpha$) for the isotropic La-doped Y$_2$O$_3$ phase, the following equation is used [48]:

$$\alpha = \frac{dL/L_0}{\Delta T}$$

Here, $\Delta T$ is the difference in temperature. As shown in Fig. 5(a), the experimental data of the thermal expansion length vs. temperature approximates to a line, implying that no phase transition occurs with an increase of temperature. It is also found that, as shown in Fig. 5(b), owing to the lattice relaxation induced by doping, the thermal expansion coefficient is generally increased upon an increase of doping concentration.

To investigate the effects of the concentrations of sintering additives on the thermal conductivity of the 5.0, 10.0, and 15.0 at% La-doped Y$_2$O$_3$ samples, the thermal conductivity is estimated as follows:

$$\kappa = \lambda \cdot C_p \cdot \rho$$

In here, $\kappa$ is the thermal conductivity, $\lambda$ is the thermal diffusivity, $C_p$ is the constant pressure heat capacity, and $\rho$ is the relative density. As the grain size is several orders of magnitude larger than the average phonon-free path in the sample, the impact of the grain size on the heat transfer is not dominant [51]. As shown in Fig. 6(a), with an increase of the concentration of doped La$^{3+}$ ions, both the thermal diffusivity and the heat capacity of the sample significantly decrease, indicating that the thermal diffusivity and heat capacity of the Y$_2$O$_3$ samples show significant dependence on the La$_2$O$_3$ doping concentration. In addition, the density increases, owing to the difference in atomic weight between Y$^{3+}$ ion (88.9) and La$^{3+}$ ion (138.9) (Fig. 6(b)). According to the method proposed in the literature [33], the thermal conductivities of the La-doped Y$_2$O$_3$ ceramics are calculated to be 7.77, 6.13, and 5.82 W/(m·K) for the samples with 5.0, 10.0, and 15.0 at% La$^{3+}$ concentrations, respectively. It should be pointed out that the thermal conductivity decreases slightly with an increase in La-doping concentration, owing to defects from doping (Fig. 6(b)).

The thermal diffusivity, heat capacity, and density of the 15.0 at% La-doped Y$_2$O$_3$ are acquired in the range of 25–800 °C, to elucidate the temperature effects on the thermal conductivity changes. As shown in Fig. 6(c), the decrease in thermal diffusivity through the increase of temperature is attributed to the strong phonon–phonon scattering [51]. In particular, as shown in Fig. 6(d), the heat capacity increases from 0.43 (25 °C) to 0.53 (800 °C), whereas the thermal conductivity decreases from 5.82 (25 °C) to 2.24 (800 °C). The 15.0 at% La-doped Y$_2$O$_3$ ceramic exhibits a superior thermal conductivity as compared to traditional laser glasses [52]. Thus, we can provide an effective and convenient method for producing high-transparency Y$_2$O$_3$ ceramics with excellent thermal and mechanical properties.

4 Conclusions

In summary, a Y$_2$O$_3$ transparent ceramic is successfully prepared via vacuum sintering while adding La$_2$O$_3$ as a
sintering aid. A sintering kinetics analysis reveals that the grain-boundary diffusion dominates the densification kinetics. The experimental results show that the diffusion in the grain boundary increases and the grain size decreases with increases of La\(^{3+}\) concentration. In the 15.0 at% La-doped Y\(_2\)O\(_3\) ceramic, a lack of pores and a uniform microstructure are acquired at a sintering temperature of 1750 °C, and the maximum in-line transmittance at a wavelength of 1100 nm reached 81.2%. Furthermore, both the mechanical and thermal properties of the transparent ceramics are improved with increases of sintering additive contents. We believe that the current study may propose a method for synthesizing high-quality laser transparent ceramics, which would be of both scientific and technological significance.

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