Transparent Er$^{3+}$-Doped Y$_2$O$_3$ Ceramics with Long Optical Coherence Lifetime

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ABSTRACT: Er$^{3+}$-doped Y$_2$O$_3$ nanoparticles (NPs) are used to synthesize transparent ceramics by hot isostatic pressing. Two sizes of NPs are studied, and 40 nm NPs show better performance than 200 nm NPs in transparent ceramics syntheses. The axial optical transmission through millimeter thickness of the prepared ceramics is about 80% in the wavelength range of 1000–2000 nm. For a sample with 11.5 ppm Er$^{3+}$, the inhomogeneous broadening of the $^4$I$_{13/2}$ to $^4$I$_{15/2}$ transition for the C$_4$ site is as low as 0.42 GHz (full width at half-maximum), and the homogeneous line width is 11.2 kHz at a temperature of 2.5 K and in a 0.65 T magnetic field. This indicates that a majority of Er$^{3+}$ ions are sitting in sites with very low structural disorder.

INTRODUCTION

Quantum memories are important components for quantum information processing, such as quantum computing and long-distance quantum communication. Among studied systems, rare-earth ion-doped solids offer interesting perspectives as quantum optical memories. The 4f–4f transitions of rare-earth ions are extremely narrow with long optical coherence time, which makes them suitable for transferring photonic quantum states to collective atomic states. However, these optical transitions are usually strongly affected by the local structural environments of dopant ions. To achieve a long optical coherence time, rare-earth ions are ideally incorporated into the crystal lattices of the host material. To date, research in this field mainly focuses on rare-earth-doped single crystals.

Transparent polycrystalline ceramics could be alternatives to single crystals in optical applications. Compared to single crystals, polycrystalline ceramics offer a number of advantages such as large-scale production and ease of processing in device fabrications. Recently, Goldner et al. have reported transparent Eu$^{3+}$-doped Y$_2$O$_3$ ceramics synthesized by sintering a mixture of Y$_2$O$_3$ nanoparticles (NPs, 50 nm) and Eu$_2$O$_3$ NPs (200 nm). After minimizing defect centers, optical studies reveal homogeneous line widths on the order of 4 kHz, which are comparable to the line widths achieved in the rare-earth-doped single crystals. This study suggests that polycrystalline ceramics are interesting alternatives to single crystals in quantum information processing.

We present here a new method to synthesize transparent Er$^{3+}$-doped Y$_2$O$_3$ ceramics using Er$^{3+}$-doped Y$_2$O$_3$ NPs as starting materials. Doping Er$^{3+}$ ions into NPs through a solution-phase synthesis can help to improve Er$^{3+}$-ion distribution in the Y$_2$O$_3$ matrix and discourage the Er$^{3+}$ clustering. Hot isostatic pressing (HIP) is used to sinter Er$^{3+}$-doped Y$_2$O$_3$ NPs into transparent ceramics. The axial optical transmission through millimeter thickness of the synthesized ceramics is about 80% in the wavelength range of 1000–2000 nm. The optical coherence lifetime of the Er$^{3+}$ $^4$I$_{13/2}$ to $^4$I$_{15/2}$ transition is measured by a two-pulse photon echo technique. In a 11.5 ppm Er$^{3+}$ sample, a 11.2 kHz homogeneous line width is recorded at a temperature of 2.5 K and in a 0.65 T magnetic field.

RESULTS AND DISCUSSION

Er$^{3+}$-doped Y$_2$O$_3$ NPs are synthesized by the reaction of urea, YCl$_3$, and ErCl$_3$ in boiled water, which produces Y$_{1-x}$Er$_x$(OH)$_2$CO$_3$H$_2$O NPs. The latter are converted into Er$^{3+}$-doped Y$_2$O$_3$ NPs by thermal annealing in air at 750 °C. The powder X-ray diffraction analysis reveals a pure cubic Y$_2$O$_3$ phase (Figure 1a). We find that the size of NPs can be tuned by simply changing the synthesis process while still using the same concentrations of chemical regents: heating up a mixture of urea (6.7 mol) and metal salts (0.22 mol) in water (4.25 L) produces NPs of about 200 nm, and pouring a urea solution (6.7 mol in 0.25 L water) into a boiled metal salt solution (0.22 mol in 4 L of water) produces NPs of about 40 nm (Figure 1b). The Er$^{3+}$ concentration in NPs can be tuned by varying the Er/Y ratios in the starting materials.

HIP is used to sinter Er$^{3+}$-doped Y$_2$O$_3$ NPs into transparent ceramics. In a typical process, Er$^{3+}$-doped Y$_2$O$_3$ NPs are pressed into a pellet in a steel die at approximately 8 klb force. This pellet is isostatically pressed at 25 kpsi in a latex isopressing sheath at room temperature, followed by sintering at 1500 °C.
in air for 2 h. The pellet is then hot isostatically pressed at 1490 °C for 16 h at 29 kpsi under an argon atmosphere in a graphite furnace. The pellet is buried inside of Y₂O₃ powder during HIP to reduce carbon contamination, which comes from the graphite furnace. Three sintering processes I–III, summarized in Table 1, are studied in this work. These processes use the same pressure (29 kpsi) and reaction time (2 h for air sintering and 16 h for HIP) but different temperatures in air sintering and HIP. To minimize the grain growth during HIP, temperatures in air sintering are slightly higher than temperatures in HIP in all three processes.

Table 1. Air Sintering and HIP Temperatures of Processes I–III

|     | air sintering temperature (°C) | HIP temperature (°C) |
|-----|-------------------------------|----------------------|
| I   | 1550                          | 1515                 |
| II  | 1500                          | 1490                 |
| III | 1470                          | 1460                 |

Figure 2a shows a pellet (1.85 mm thick and 10 mm diameter) prepared using 40 nm Er³⁺-doped Y₂O₃ NPs by process II. The sample is polished at a surface finish of 0.5 μm (Rₐ = \( \frac{1}{n} \sum |R_{ai}| \)); the arithmetic average of roughness). The axial optical transmission through the thickness of the pellet is about 80% in the wavelength range of 1000–2000 nm (Figure 2b), which is close to the theoretical limit of Y₂O₃ at 1535 nm (82.7%) when only considering the surface reflections. The grain boundary in this pellet is too small to be imaged by SEM, which has a resolution of 3 nm. Electron backscatter diffraction (EBSD) is used to characterize the grains (Figure 2c). The grain sizes (the equivalent diameters) range from 0.18 to 1.7 μm, with an average area of 0.31 μm² (analysis of 1500 grains).

The grain size can be tuned by varying the sintering temperatures. In a pellet prepared by process I at a higher temperature (Table 1), the grain sizes range from 0.7 to 2.1 μm, with an average area of 0.90 μm² (analysis of 750 grains).

The transmitted scattering of Er³⁺-doped Y₂O₃ pellets prepared by different sintering processes was also studied. When light passes through the pellets, the intensity of incident beam is mainly attenuated by absorption, scattering, and surface loss. The difference in scattering can be related to the size and density of scattering centers (such as residual pores) in different samples. The transmitted scattering coefficient (μ, dB/mm) is estimated by

\[ \mu = 10 \times \log\left(\frac{T_t}{T_a}\right)/L \]

where Tₜ, Tₐ, and L are the total transmission, the axial transmission, and the thickness of the pellet, respectively. Figure 3 summarizes the transmitted scattering coefficients of

Figure 3. Transmitted scattering coefficient of Er³⁺-doped Y₂O₃ ceramic pellets prepared by processes I–III using 40 nm NPs.

Figure 1. (a) Powder X-ray diffraction pattern of Er³⁺-doped Y₂O₃ NPs (200 nm). The red stick pattern corresponds to PDF 00-041-1105 (cubic Y₂O₃). (b,c) Scanning electron microscopy (SEM) images of Er³⁺-doped Y₂O₃ NPs synthesized using the same concentrations of chemical reagents by (a) heating up a mixture of urea (6.7 mol) and metal salts (0.22 mol) in H₂O (4.25 L) solution and (b) pouring a urea solution (6.7 mol in 0.25 L water) into a boiled metal salt solution (0.22 mol in 4 L water).
the pellets prepared using 40 nm NPs by sintering processes I−III. In the long-wavelength range of over 1000 nm, the three samples show very low scattering, and the transmitted scattering coefficients are close to zero. This indicates that the density of large-size scattering centers (such as micrometer pores) is very low in our ceramic pellets. In the visible range (<750 nm), however, scattering in all three samples is stronger. Among processes I−III, the lower temperature process produces ceramic pellets with the least scattering.

The NP size also influences the ceramics synthesis. When 40 nm NPs are used, all processes I−III produce ceramics with good transparency. However, only process I, which has the highest temperature among processes I−III, can produce transparent ceramics by using 200 nm NPs (Figure 4a,b). Using the same process I, ceramics produced by 40 nm NPs have lower scattering than ceramics produced by 200 nm NPs (Figure 4c). These results indicate that, in our method, smaller size NPs work better in the synthesis of transparent ceramics.

In the following optical characterizations, three Er3+-doped Y2O3 ceramic samples prepared by process I (Table 1; 1550 °C air sintering followed by 1515 °C HIP) with different Er3+ concentrations are studied. Er3+ concentrations are measured by inductively coupled plasma mass spectrometry.

The inhomogeneous line width ($\Gamma_{\text{inh}}$) of transition from the lowest state in 4I15/2 to the lowest state in 4I13/2 for C2 sites around 1535 nm is magnetic-field-dependent owing to the fact that random orientation of the crystalline phase causes inhomogeneous Zeeman splitting. Thus, the intrinsic inhomogeneous line width is measured at zero field and at low temperature (1.7 K) to reduce phonon broadening. The line width is below the instrument resolution of our grating-based optical spectral analyzer (Ando AQ6317B) (Figure 5a). Thus, we use piezo-scanned laser to measure the transmission directly (for details, see Methods section). The transmission curve for a sample with an Er3+ concentration of 11.5 ppm is shown in Figure 5b and fitted to a Lorentzian function. The inhomogeneous broadening [full width at half-maximum (fwhm)] is 0.42 ± 0.02 GHz. This strongly indicates that the majority of Er3+ ions is sitting in the crystalline phase with very little structural disorder.

The homogeneous line width ($\Gamma_h$) of the Er3+ 4I13/2−4I15/2 transition at 1534.94 nm (C2 line) in transparent Er3+-doped Y2O3 ceramics is measured by a two-pulse photon echo spectroscopy19 on three samples with different Er3+ concentrations. A typical data set is shown in Figure 6 for the 11.5 ppm sample. Echo intensity is fitted to an exponential function $I(t_{12}) = I_0 \exp[-4t_{12}/T_2^*]$. The homogeneous line width is calculated as $\Gamma_h = 1/\pi T_2^*$. Here, we saw relatively small

Figure 4. (a,b) Er3+-doped Y2O3 ceramic pellets prepared using 200 nm NPs by processes I (a) and II (b). (c) Transmitted scattering coefficient of Er3+-doped Y2O3 ceramic pellets prepared by process I using 40 and 200 nm NPs.

Figure 5. (a) Transmission spectrum measured using our grating-based optical spectrum analyzer. The longer period oscillation comes from the multiple reflections from the cryostat windows. The line width is instrument-resolution-limited. (b) Transmission spectrum measured using the laser scanning technique. The spectrum is fitted to a Lorentzian function with fwhm equal to 0.42 ± 0.02 GHz.

Figure 6. Photon echo intensity as a function of the delay $t_{12}$ (μs) at different temperatures for the 11.5 ppm sample. Traces are fitted to the exponential decay function to extract the dephasing time.
contribution from the spectral diffusion, likely because of the low Er\(^{3+}\) concentration.

The temperature dependence of the homogeneous line width (11.5 ppm sample) at 0.65 T is shown in Figure 7a. Above 4 K, we see the typical \( T \) dependence because of the Raman process.\(^{20} \) At lower temperature, there is an additional \( \Gamma (T) = a T^c \), with \( a = 4.5 \) kHz/K contribution that likely results from the residue disorder in the sample.\(^{16,21~24} \)

Magnetic field dependence of the homogeneous line width of the same sample at \( T = 1.5 \) K is shown in Figure 7b. The line width drops precipitously with the initial small magnetic field owing to removal of the degeneracy of the Zeeman sublevels and subsequent decrease in the electron population in the upper-ground-state Zeeman level. As the magnetic field increases, the spin flip rate starts to increase because of the increase in the available phonon density of states.\(^{25} \) This resulted in a small “bump” for the homogeneous line width at an intermediate magnetic field. Similar behavior is observed in various Er\(^{3+}\)-doped crystals and glasses.\(^{16,25~27} \)

Homogeneous line widths for three samples with different Er\(^{3+}\) concentrations are summarized in Table 2. Sample with high Er\(^{3+}\) concentration has a large homogeneous line width owing to Er\(^{3+}\)–Er\(^{3+}\) spin interaction. As the concentration gets lower, the average Er\(^{3+}\) ion distance gets larger. Below 10 ppm, the Er\(^{3+}\) concentration has negligible effect on the homogeneous line width. The homogeneous line width of a 1.15 ppm sample at 2.5 K, 0.65 T is 11.2 ± 0.2 kHz (corresponding to \( T_2 = 28 \) \( \mu \)s). This result is comparable to the reported line width of 50 ppm Er\(^{3+}\)-doped \( \text{Y}_2\text{O}_3 \) single crystals.\(^{6} \)

## CONCLUSIONS

We report the development of Er\(^{3+}\)-doped \( \text{Y}_2\text{O}_3 \) transparent ceramics with long optical coherence lifetime. Low-porosity transparent ceramics are prepared by sintering NPs using HIP. Er\(^{3+}\)-doped \( \text{Y}_2\text{O}_3 \) NPs synthesized by a wet-chemistry approach are used as starting materials to improve the Er\(^{3+}\) dispersion. The prepared Er\(^{3+}\)-doped \( \text{Y}_2\text{O}_3 \) ceramics show good optical transparency with about 80% axial transmission through 1.85 mm thickness in the wavelength range of 1000–2000 nm. The very small inhomogeneous broadening (0.42 GHz) and homogeneous broadening (11.2 kHz) of the Er\(^{3+}\) \( ^{4}\text{I}_{15/2} \rightarrow ^{4}\text{I}_{15/2} \) transition in our ceramics strongly suggest that Er\(^{3+}\) ions reside in the crystalline phase with low structural disorder. Such transparent ceramics with well-controlled Er\(^{3+}\) doping are promising materials for quantum memory applications.

## METHODS

### Materials

YCl\(_3\)·6H\(_2\)O (99.9%), ErCl\(_3\)·6H\(_2\)O (99.995%), and urea (99.0–100.5%) were purchased from Aldrich and used as received.

**Er\(^{3+}\)-Doped \( \text{Y}_2\text{O}_3 \) NP Synthesis.** NPs of 200 and 40 nm were synthesized by different processes while using the same concentrations of chemical regents. NPs of 200 nm were synthesized by heating a mixture of YCl\(_3\)·6H\(_2\)O/ErCl\(_3\)·6H\(_2\)O (in total 0.22 mol) and urea (6.7 mol) in deionized (DI) water (4.25 L) to 100 °C and keeping at 100 °C for 1 h. The thermal decomposition of urea provides OH\(^-\) and CO\(_3^{2-}\), which react with metal ions and produce \( \text{Y}_{1-x}\text{Er}_x\text{(OH)}\text{CO}_3\text{H}_2\text{O} \) NPs. \( \text{Y}_{1-x}\text{Er}_x\text{(OH)}\text{CO}_3\text{H}_2\text{O} \) NPs were collected by filtration and were converted into \( (\text{Y}_{1-x}\text{Er}_x)_2\text{O}_3 \) NCs by annealing at 750 °C for 90 min. The chemical yield was about 95%. Er\(^{3+}\)-doped \( \text{Y}_2\text{O}_3 \) NPs of 40 nm were synthesized by pouring a urea solution (6.7 mol in 0.25 L DI water) into a boiled YCl\(_3\)·6H\(_2\)O/ErCl\(_3\)·6H\(_2\)O solution (0.22 mol in 4 L water), and the reaction was allowed to run for 1 h. \( \text{Y}_{1-x}\text{Er}_x\text{(OH)}\text{CO}_3\text{H}_2\text{O} \) NPs were collected by a filtration process and were converted into \( (\text{Y}_{1-x}\text{Er}_x)_2\text{O}_3 \) NCs by annealing at 750 °C for 90 min. The chemical yield was about 95%.

**Er\(^{3+}\)-Doped \( \text{Y}_2\text{O}_3 \) Transparent Ceramics Synthesis.** Millimeter thick Er\(^{3+}\)-doped \( \text{Y}_2\text{O}_3 \) transparent ceramic pellets were prepared by HIP. In a typical process, Er\(^{3+}\)-doped \( \text{Y}_2\text{O}_3 \) NPs were pressed into a pellet in a 3/4 in. steel die at approximately 8 kbar force. This pellet was isostatically pressed at 25 kpsi in a latex isopressing sheath at room temperature, followed by sintering at 1550 °C in air for 2 h. The pellet was then pressed at 1515 °C for 8 h at 29 kpsi in an argon atmosphere in a graphite furnace using HIP. To reduce carbon contamination from the graphite furnace, the pellet was buried in \( \text{Y}_2\text{O}_3 \) powder during HIP.

**SEM Characterizations.** SEM observation was conducted with a Zeiss 1550 VP field-emission scanning electron microscope. The SEM samples were prepared by putting a drop of NP–water suspensions on the surface of 300 mesh copper transmission electron microscopy (TEM) grids coated with lacey carbon film. The NP-decorated lacey carbon TEM grids were fixed to a SEM stub using conductive carbon tape and analyzed without applying a conductive coating. The Zeiss

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Table 2. Homogeneous Line Width for Er\(^{3+}\)-Doped \( \text{Y}_2\text{O}_3 \) Ceramics with Different Er\(^{3+}\) Concentrations

| Er\(^{3+}\) (ppm) | \( \Gamma_h \) (kHz) @ 0.65 T, 2.5 K |
|-----------------|----------------------------------|
| 2.0             | 11.4 ± 0.7                       |
| 11.5            | 11.2 ± 0.2                       |
| 162             | 49 ± 5                           |

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Figure 7. (a) Temperature dependence of the homogeneous line width \( \Gamma_h \) (\( B = 0.65 \) T). At high temperature (>4 K), the line width is dominated by the two phonon Raman process; at low temperature, the major contribution shows a \( T \) temperature dependence and likely comes from tunneling states. (b) Magnetic field dependence of the homogeneous line width \( \Gamma_h \) (\( T = 1.5 \) K).
SmartSEM instrument control software was used to measure particle diameter.

**EBSD Analysis.** EBSD analysis was done on a Hitachi SU-70 FE-SEM equipped with an Oxford EDS/EBSD system (Oxford/AZTEC software with a Nordlys camera). A piece of the material was removed and mounted in epoxy. The material was then polished to yield a surface quality finish that would allow diffraction pattern generation. For data acquisition, the sample was placed in a SEM and tilted at a 70° angle to generate Kikuchi patterns that were collected on a camera and then analyzed. Post-process done on the data included extrapolations of wild spikes and zero solutions in the data. Pseudosymmetry around the (111) axis was corrected before grain size was determined.

**Optical Inhomogeneous Broadening Measurement.**

The inhomogeneous line width ($\Gamma_{\text{inh}}$) was measured using a piezo-scanned laser (TOPTICA DLpro). Briefly, the laser was scanned using a saw tooth modulation with a peak to peak of 15 V at 2 Hz. The transmission power was monitored with a fast photodiode and recorded on an oscilloscope. The frequency conversion of the piezodrive was independently fast photodiode and recorded on an oscilloscope. The source was a tunable diode laser (TOPTICA DLpro) with <50 kHz line width. The light was temporally shaped by an acoustic-phonon modulator (pulse width 50 ns), before being amplified by an electro-optic modulator and detected through a second electro-optic modulator and detected on a nanosecond oscilloscope. The output was controlled so that there was no gain depletion seen from pulse to pulse. The input peak intensity was carefully controlled so that there was no gain depletion seen from pulse to pulse. The output was filtered through a 1 nm pass-band filter and an electro-optic modulator (pulse width 50 ns) to cut off amplified spontaneous emission from the amplifier. The beam was focused down to a spot of ~50 μm. The input peak intensity on the sample was 2 kW/cm². The resulting echo was filtered through a second electro-optic modulator and detected by an InGaAs avalanche photodiode and averaged on the oscilloscope. An active background subtraction was implemented by removing the first pump pulse in every other pulse train, and the signal was subtracted from the background. All delays between the pulses were synchronized by a personal computer.

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