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Highly efficient lasing and thermal properties of Tm:Y₂O₃ and Tm:(Y,Sc)₂O₃ ceramics

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We report on thermal, spectroscopic and laser properties of transparent 5 at.% Tm³⁺-doped yttria and "mixed" yttria-scandia ceramics fabricated by vacuum sintering at 1750 °C using nanoparticles produced by laser ablation. The solid-solution (Tm_{0.05}Y_{0.698}Sc_{0.252})₂O₃ ceramic features a broadband emission extending up to 2.3 µm (gain bandwidth: 167 nm) and high thermal conductivity of 4.48 Wm⁻¹K⁻¹. A Tm:Y₂O₃ ceramic laser generated 812 mW at 2.05 µm with a slope efficiency η of 70.2%. For the Tm:(Y,Sc)₂O₃ ceramic, the output power was 523 mW at 2.09 µm with η = 44.7%. These results represent recordhigh slope efficiencies for any parent or "mixed" Tm³⁺doped sesquioxide ceramics. © 2023 Optical Society of America

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Thulium ions (Tm³⁺, electronic configuration: [Xe]4f¹²) are known for their broadband emission in the eye-safe spectral range around 2 µm due to the ${}^{3}F_{4} \rightarrow {}^{3}H_{6}$ electronic transition [1]. Tm³⁺ ions offer intense absorption at 0.8 µm (the spectral range covered by AlGaAs diode lasers) and a cross-relaxation (CR) process, ${}^{3}H_{4} + {}^{3}H_{6} \rightarrow {}^{3}F_{4} + {}^{3}F_{4}$, leading to a pump quantum efficiency up to 2 (a one-to-two pump process) [2]. Due to the large Stark splitting of the ground-state (${}^{3}H_{6}$, 788 cm⁻¹ for Tm:Y₂O₃ [3]), Tm³⁺-doped crystals are extremely suitable for broadly tunable and mode-locked lasers [4,5].

Among the host matrices for Tm³⁺ doping, cubic (sp. gr. *Ia* $\overline{3}$, Ctype) rare-earth sesquioxides A₂O₃ (A = Y, Lu, Sc) appear attractive because of a combination of good thermal conductivity of the host matrix (κ = 12.7 Wm⁻¹K⁻¹ for Y₂O₃) and advantageous spectroscopic properties of the dopant ions, i.e., broad emission spectra extending well above 2 µm owing to strong crystal-fields, long upper laser level luminescence lifetimes and efficient CR [1]. Highly-efficient and power-scalable Tm:A₂O₃ lasers at ~2 µm are known [1,6,7]. Loiko *et al.* reported on a diode-pumped Tm:Lu₂O₃ crystal-rod laser generating 47.5 W at 2.1 µm with a slope efficiency of 59% [1]. However, the growth of Tm: A_2O_3 single-crystals is complicated by their high melting points (2425 °C for Y_2O_3) which are withstood only by Rh crucibles [8].

Transparent ceramics are polycrystalline materials composed of closely packed single-crystalline grains with a random orientation featuring a weak light scattering under a low density of pores and a lack of secondary phases. The ceramic technology is well applicable to cubic sesquioxides [7,9,10] offering lower synthesis temperature (~1750 °C), well-preserved spectroscopic behavior of the dopant ions, good thermo-mechanical properties (under proper synthesis conditions) and well-controlled doping. Tm:A₂O₃ laser ceramics are known [7,11-13]. Wang *et al.* reported on a diode-pumped 2 at.% Tm:Y₂O₃ ceramic laser delivering 7.25 W at 2.05 μ m with a slope efficiency of 40% exceeding the Stokes limit [13].

Apart from the parent sesquioxides, their binary solid-solutions $Tm:(A_{1x}B_x)_2O_3$ attract attention for generation of ultrashort pulses from mode-locked lasers [5,14]. This is due to an inhomogeneous spectral broadening in such compounds. Zhao *et al.* reported on a mode-locked Tm:LuYO₃ ceramic laser delivering pulses as short as 57 fs at 2045 nm, i.e., 8 optical cycles [5]. The ceramic technology is suited for the precise control of the A/B balances in such materials.

In the present work, we report on the thermal and spectroscopic properties and highly-efficient $\sim 2 \ \mu m$ laser operation of Tm:Y₂O₃ and Tm:(Y,Sc)₂O₃ transparent sesquioxide ceramics fabricated by an original method employing laser-ablated nanoparticles.

Transparent Tm: $(Y_{1-x}Sc_x)_2O_3$ ceramics were fabricated by solidstate consolidation of nanoparticles synthesized by laser ablation of a solid target material [15]. To prepare the targets, commercial high-purity A_2O_3 (A = Y, Sc, Tm) powders were dry-mixed for 24 h in the proportion Tm₀₀₅(Y_{1-x}Sc_x)₁₉₅O₃ (x = 0, 0.125, 0.25). A small amount of ZrO₂ (1.7 mol%) acting as a grain growth inhibitor was used [16]. The mixtures were compacted and sintered at 1100 °C for 5 h in air. The vapor phase above the target surface formed under irradiation by a pulsed CO₂ laser was cooled by an air flow to condense and crystallize as nanoparticles. Their composition was (Tm₀₀₅Y_{0.95})₂O₃, (Tm₀₀₅Y_{0.829}Sc_{0.121})₂O₃ and (Tm₀₀₅Y_{0.698}Sc_{0.252})₂O₃

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uniaxially pressed in a stainless-steel die at 200 MPa to form green bodies, which were sintered at 1750 °C for 5 h under a residual gas pressure of 10^{-3} Pa. The sintered ceramics were annealed at 1400 °C for 2 h in air. The ceramic disks (Φ 12 mm, ~1.8-1.9 mm-thick) were polished to laser-grade quality on both sides. A photograph of annealed polished ceramic disks is shown in Fig. 1(a). They were transparent with a slight yellow coloration due to Tm³⁺ doping.



Fig. 1. 5 at.% Tm: $(Y_{1*}Sc_x)_2O_3$ ceramics: (a) a photograph of laser-grade polished ceramic disks; (b) a typical SEM image of an etched surface of the $(Tm_{005}Y_{0.829}Sc_{0.121})_2O_3$ ceramic.

The ceramics were of single-phase nature (sp. gr. $Ia\overline{3}$, bixbyite structure). A typical SEM image of a thermally etched surface of the $(Tm_{0.05}Y_{0.829}Sc_{0.121})_2O_3$ ceramic is shown in Fig. 1(b). The samples consisted of polyhedral-shaped micro-crystallites separated by well-defined and clean boundaries. Partial substitution of Y³⁺ by Sc³⁺ accelerated the diffusion processes and caused a notable increase in the grain size ($D_{grain} = 6.8, 21.2$ and $28.2 \,\mu\text{m}$ for 0, 12.1 and 25.2 at.% Sc³⁺). The content of μm -sized pores progressively increased, $X_{pore} = 0.6, 25.9$ and 46.1 ppm, respectively. It is likely that the accelerated grain growth led to entrapment of a considerable fraction of pores. The transmission of the ceramic disks at ~1 μm was T = 81.5%, 81.0% and 80.5%, respectively, close to the theoretical limit.



Fig. 2. 5 at.% Tm:($Y_{1:x}Sc_x$)₂O₃ ceramics: temperature dependence of (a) specific heat C_{p_r} (b) thermal diffusivity α and (c) thermal conductivity κ ; (d) RT thermal conductivity for (Tm_{0.05}Y_{1:x}Sc_x)₂O₃ and (Y_{1:x}Yb_x)₂O₃ [17] ceramics, *points* – experiment, *curves* – simulation (Gaume model).

The thermophysical characteristics of ceramics were studied by the laser flash method using a NETZSCH LFA 467 HyperFlash at 25 – 350 °C under Ar atmosphere. The samples were covered with a graphite layer. The thermal conductivity was calculated as $\kappa = \alpha C_p \rho$, where α is the thermal diffusivity, C_p is the specific heat and ρ is the density evaluated from the XRD analysis, see Fig. 2(a-c). At room temperature (RT), κ gradually decreases from 5.93 to 4.48 Wm⁻¹K⁻¹ when raising the Sc³⁺ content from 0 to 25.2 at.%. The concentration dependence of κ for (Tm_{0.05}Y_{1-x}Sc_x)₂O₃ and (Y_{1-x}Yb_x)₂O₃ [17] ceramics is well explained using the Gaume model [18], Fig. 2(d).

The formation of a substitutional solid-solution for Sc-containing ceramics was confirmed by Raman spectroscopy, Fig. 3. The most intense Raman band assigned to $F_g + A_g$ modes of the cubic bixbyite structure broadens and experiences a progressive shift towards higher frequencies with increasing the Sc³⁺ content. For 25.2 at.% Sc³⁺, it is centered at 390 cm⁻¹ (bandwidth: 31 cm⁻¹) and the highest phonon energy is 614 cm⁻¹.



Fig. 3. Raman spectra of 5 at.% Tm: $(Y_{1-x}Sc_x)_2O_3$ ceramics, $\lambda_{exc} = 514$ nm, *numbers* – Raman frequencies in cm⁻¹.



Fig. 4. Spectroscopy of Tm³⁺ ions in (Y_{1-x}Sc_x)₂O₃ ceramics (x = 0 - 0.252): (a) absorption cross-sections, σ_{abs} , for the ³H₆ \rightarrow ³H₄ transition; (b) SE cross-sections, σ_{SE} , for the ³F₄ \rightarrow ³H₆ transition; (c) luminescence decay curves from the ³F₄ manifold, $\lambda_{exc} = 1630$ nm, $\lambda_{lum} = 1960$ nm, powdered samples, τ_{lum} – luminescence lifetime; (d) gain cross-sections, $\sigma_{gain} = \beta\sigma_{SE} - (1 - \beta)\sigma_{abs}$, $\beta = N_2(^3F_4)/N_{Tm}$ - inversion ratio. *Arrows* indicate the pump and laser wavelengths.

Figure 4(a) shows the absorption cross-sections, σ_{abs} , for the ${}^{3}H_{6} \rightarrow {}^{3}H_{4} \text{ Tm}{}^{3+}$ transition. The Tm ${}^{3+}$ ion density N_{Tm} was 1.35, 1.38 and

1.42 [10²¹ at/cm³] for 0, 12.1 and 25.2 at.% Sc³⁺, respectively. By adding Sc³⁺, the absorption spectra become less structured. For the $(Tm_{0.05}Y_{0.698}Sc_{0.252})_2O_3$ ceramic, the maximum σ_{abs} is 0.31×10^{-20} cm² at 795.3 nm corresponding to an absorption bandwidth (FWHM) of 25 nm. The stimulated-emission (SE) cross-section spectra for the ${}^{3}F_{4} \rightarrow {}^{3}H_{6}$ transition are plotted in Fig. 4(b). By replacing Y³⁺ cations $(R_{\rm Y} = 0.9 \text{ Å for VI-fold } O^2 \text{ coordination})$ with much smaller Sc³⁺ ones $(R_{\rm Sc} = 0.745 \text{ Å})$, the emission spectra of Tm³⁺ ions broadened and shifted towards longer wavelengths due to increased crystal-field strength in scandia as compared to yttria. In the spectral range where laser operation is expected for the (Tm0.05Y0.698Sc0.252)2O3 ceramic, $\sigma_{\text{SE}} = 0.24 \times 10^{-20} \text{ cm}^2$ at 2075.6 nm. The reabsorption-free upper laser level (3F4) lifetime of Tm3+ ions was weakly dependent on the Sc³⁺ content, τ_{lum} = 2.41 – 2.55 ms, Fig. 4(c). The gain crosssections, $\sigma_{gain} = \beta \sigma_{SE} - (1 - \beta) \sigma_{abs}$, for the ${}^{3}F_{4} \leftrightarrow {}^{3}H_{6}$ transition were calculated. For $(Tm_{0.05}Y_{0.698}Sc_{0.252})_2O_3$, for small inversion ratios $\beta <$ 0.06, a local peak at \sim 2.09 μ m dominates in the spectra and for higher β , another peak at ~1.95 µm appears. For small β = 0.06, the gain bandwidth (FWHM) reaches 167 nm indicating a great potential for ultrashort (sub-100 fs) pulse generation.

The inhomogeneous spectral broadening of Tm^{3+} absorption / emission bands in "mixed" (solid-solution) ceramics was confirmed by low temperature (12 K) spectroscopy of the ${}^{3}F_{4} \leftrightarrow {}^{3}H_{6}$ transition, Fig. 5. An additional spectral broadening as compared to $Tm:Y_{2}O_{3}$ single-crystals was induced by ZrO₂.



Fig. 5. Low-temperature (LT, 12 K) spectroscopy of Tm^{3+} ions in the Y_2O_3 and $(Y,Sc)_2O_3$ ceramics: (a) the ${}^{3}H_6 \rightarrow {}^{3}F_4$ transition in absorption; (b) the ${}^{3}F_4 \rightarrow {}^{3}H_6$ transition in emission. ZPL – zero-phonon line.

For laser experiments, we employed the (Tm0.05Y0.698Sc0.252)2O3 and $(Tm_{0.05}Y_{0.95})_2O_3$ ceramics. The laser elements were cut from the central part of the ceramic disks, they were double-side polished to laser-grade quality with good parallelism and left uncoated. They were fixed on a passively cooled Cu-holder using a silver paint. A hemispherical cavity was formed by a flat pump mirror (PM) coated for high transmission (HT, T = 78.7%) at 0.81 µm and high reflection (HR, R > 99.9%) at 1.86 – 2.33 µm and a concave (radius of curvature: -100 mm) output coupler (OC) with a transmission T_{OC} in the range of 0.2% - 3.5% at the laser wavelength. The total cavity length was 102 mm. The laser element was placed near the PM with an airgap of <1 mm. The pump source was a Ti:Sapphire laser (3900S, Spectra Physics) delivering up to 4.0 W at 811 nm with a nearly diffraction limited beam ($M^2 \sim 1$). The pump radiation was focused into the ceramic through the PM using an AR-coated aspherical lens (f = 75 mm). The pump spot diameter in the focus was 50±10 µm. The pumping was in double-pass due to a backreflection from the OCs at the pump wavelength ($R \sim 80\%$). For the $(Tm_{0.05}Y_{0.95})_2O_3$ ceramic and the optimum $T_{0C} = 3.5\%$, the total pump absorption under lasing conditions was 51.0% representing certain ground-state (${}^{3}H_{6}$) bleaching. The radius of the laser mode in the ceramic was calculated using the ABCD method accounting for the thermal lens in Tm:Y₂O₃, $w_{L} = 57\pm5$ µm, indicating a good mode-matching. The spectra of laser emission were measured using an optical spectrum analyzer (Yokogawa, AQ6376).

The (Tm0.05Y0.95)2O3 ceramic delivered a maximum output power of 812 mW with a high slope efficiency η of 70.2% (vs. the absorbed pump power) for T_{0C} = 3.5%, Fig. 6(a). With increasing the output coupling, the laser threshold gradually increased from 90 mW (Toc = 0.2%) to 393 mW (T_{0C} = 3.5%). The emission wavelength experienced a slight blue-shift with increasing the output coupling, from 2061–2085 nm (T_{0C} = 0.2%) to 2048–2063 nm (T_{0C} = 3.5%) owing to the quasi-three-level nature of the ${}^{3}F_{4} \rightarrow {}^{3}H_{6}$ transition with reabsorption, Fig. 6(c). The obtained laser slope efficiency exceeds the Stokes limit, $\eta_{SLL} = \lambda_P / \lambda_L = 39.6\%$, owing to an efficient CR among adiacent Tm³⁺ ions. After Loiko *et al.* [1], for 5 at.% Tm³⁺ doping in Y_2O_3 , the quantum efficiency of excitation into the ${}^{3}F_4$ manifold is η_q = 1.97±0.03 (almost ideal one-to-two process). The upper limit for the slope efficiency is then [2] $\eta < \eta_{St,L}\eta_{q}\eta_{mode}\eta_{OC} = 69\pm3\%$, where η_{OC} $= \ln[1 - T_{0C}]/\ln[(1 - T_{0C})(1 - L)] = 87\pm3\%$ is the output-coupling efficiency (*L* is the round-trip passive loss, see below) and $\eta_{\text{mode}} \approx 1$ is the mode-matching one.



Fig. 6. ~2 µm laser performance of (a,c) $(Tm_{0.05}Y_{0.95})_2O_3$ and (b,d) $(Tm_{0.05}Y_{0.698}Sc_{0.252})_2O_3$ ceramics: (a,b) input-output dependences, η – slope efficiency; (c,d) typical spectra of laser emission.

For the yttria-scandia (Tm₀₀₅Y_{0.698}Sc_{0.252})₂O₃ ceramic, the laser generated 523 mW at 2065 – 2097 nm with a lower η of 44.7% and a laser threshold of 275 mW (for T_{0C} = 1.5%), Fig. 6(c). The laser threshold was in the range 165 – 484 mW for output coupler transmissions between 0.3% and 3.5%. The lower slope efficiency for the Sc-containing ceramic is related to higher passive losses caused by the residual porosity. The laser emission spectra for the "mixed" ceramic are shown in Fig. 6(d). For $T_{0C} \le 1.5\%$, the laser operated at 2.08 µm and for higher output coupling, additional lines at 1.96 and 1.99 µm appeared. This behavior agrees with the gain spectra for increased inversion ratios, cf. Fig. 4(d). A clear red-shift of the laser wavelength due to the Sc³⁺ effect on the emission spectra was observed.

The round-trip passive losses in the ceramics were evaluated using the Findlay-Clay analysis [19] by plotting the laser threshold $P_{\rm th}$ versus $\ln(1/R_{\rm OC})$ where $R_{\rm OC} = 1 - T_{\rm OC}$ is the output coupler reflectivity, Fig. 7. This analysis yielded $L = 0.5 \pm 0.2\%$ and $1.5 \pm 0.3\%$ for the yttria and yttria-scandia ceramics, respectively. The increase in the passive losses for the Sc-containing ceramic agrees with the higher content of pores for this material.



Fig. 7. The Findlay-Clay analysis for estimating the round-trip passive loss *L* for $(Tm_{0.05}Y_{0.05})_2O_3$ and $(Tm_{0.05}Y_{0.698}Sc_{0.252})_2O_3$ ceramics.

Suzuki *et al.* studied (Tm₀₀₃₁Y_{0.474}Sc_{0.495})₂O₃ single-crystals grown by the Czochralski method at a reduced temperature of 2100 °C. Under diode-pumping at 780 nm, an output power of 422 mW was generated at 2.11 µm with η = 45% [20]. Jing *et al.* reported on a diode-pumped (Tm_{0.048}Lu_{0.635}Sc_{0.317})₂O₃ ceramic laser delivering 1.01 W at 2.10 µm with lower η = 24% [12]. Pirri *et al.* reported on a quasi-CW diode-pumped (Tm_{0.05}Sc_{0.252}Y_{0.698})₂O₃ ceramic laser generating 1.24 W at 2.08 µm with lower η =9.5% [21]. Our results for the Tm:(Y,Sc)₂O₃ ceramic in terms of laser slope efficiency are superior to all previously studied Tm³⁺-doped "mixed" sesquioxide ceramics and close to the best results for single-crystals.

The best result for the parent Tm:Y₂O₃ ceramic was achieved by Wang *et al.*: a diode-pumped (Tm_{0.02}Y_{0.98})₂O₃ laser generated 7.25 W at 2.05 μ m with η = 40% [13]. The slope efficiency achieved in this work with the Tm:Y₂O₃ ceramic is close to the best results reported for single-crystalline Tm³⁺-doped sesquioxides [1,22].

To conclude, we report on highly-efficient $\sim 2 \,\mu m$ laser operation of Tm:Y₂O₃ and Tm:(Y,Sc)₂O₃ transparent sesquioxide ceramics. For the both parent and solid-solution ceramics, the achieved laser slope efficiencies (70.2% and 44.7%) represent record-high values for any Tm³⁺-doped sesquioxide ceramics being comparable with the best results for the corresponding single-crystals. It is due to an efficient cross-relaxation under high Tm³⁺ doping level (5 at.%) and relatively low passive losses even for "mixed" compounds owing to an optimized synthesis procedure involving Tm:(Y1-xScx)2O3 laserablated nanoparticles with a mixture of cations at the atomic level and ZrO₂ as a grain growth inhibitor. The high thermal conductivity of ceramics paves the way towards power scaling under pumping by AlGaAs laser diodes at 0.8 µm or Raman-shifted Erbium fiber lasers at 1.68 µm (in-band pumping). The laser efficiency for "mixed" ceramics could be improved by reducing their residual porosity through optimization of the processing conditions and purification of the Sc₂O₃ raw powder. The solid-solution Tm:(Y,Sc)₂O₃ ceramics feature inhomogeneously broadened emission spectra extending up to 2.3 µm making them promising for sub-100 fs pulse generation from SESAM or Kerr-lens mode-locked lasers.

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Data availability. Data underlying the results presented in this paper are not publicly available at this time but may be obtained from the authors upon reasonable request.

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