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Scandium ion doped yttrium oxide transparent ceramic from nitrate alanine microwave combustion synthesized nanopowders

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Abstract

One atomic percent Neodymium ion doped Yttrium oxide, with 25 at% scandium ion ($Nd_{0.02}Sc_{0.5}Y_{1.48}O_3$), was synthesized by nitrate alanine microwave gel combustion followed by calcinations at 1000 °C for 2 h. Phase purity of nanopowder was characterized by X-Ray Diffraction (XRD). Neodymium and scandium ion doping was confirmed by cell parameter calculation and Scanning Electron Microscope-Electron Dispersive X-ray (SEM-EDX) analysis. Particles with size range 25–35 nm with close to spherical shape were obtained as observed by Transmission Electron Microscopy (TEM). Powder on compaction followed by vacuum sintering at 1765 °C for 40 min led to the formation of ceramic with 76% transmission at 2500 nm compared to translucent ceramic obtained without scandium ion doping. This indicates formation of highly sinterable neodymium doped yttrium oxide nanopowders by nitrate alanine microwave gel combustion route with scandium ion additive. Further the absorption and emission bands of $Nd_{0.02}Sc_{0.5}Y_{1.48}O_3$ are inhomogeneously broadened and fluorescence lifetime is longer than $Nd_{0.02}Y_{1.98}O_3$.
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1. Introduction

Rare earth ion doped yttrium Y_2O_3, a C-type sesquioxide of a cubic crystal, is an ideal laser host media for its good optical, thermal, chemical, and mechanical properties [1,2]. However, it is extremely difficult to grow a high-quality Y_2O_3 crystal because of its high melting point (about 2430 °C) and phase transition just before the melting point [3,4].

Development of ceramic technology is a boon in laser material science when it comes to Y_2O_3 lasers. Originally pressure less sintering in hydrogen atmosphere was carried out of the yttrium oxide powders compact, without or with additives such as ThO_2, ZrO_2, LaO_2 or HfO_2, at very high temperature from 2000 to 2270 °C [5–9]. The other more recent is nanotechnology vacuum sintering (NTVS) [10,11].

Recent trend in ceramic processing of sesquioxides involves addition of homovalent (Sc^{+3}, Y^{+3} and La^{+3}) and heterovalent (Zr^{+4}) ions for further improving ceramic properties viz minimizing sintered ceramic grain size and broadening of emission bands [12–15] on either vacuum sintering at 1700–1950 °C for 10–20 h or by high temperature sintering in hydrogen atmosphere for up to 45 h. Scandium ion doped yttrium oxide nanopowders have been synthesized by combination of coprecipitation and solid state method, which gave 68% transmission in visible range on vacuum sintering at 1750–1850 °C [16], and by solid state reaction [17], which was sintered in H_2 atmosphere at very high temperature of 1600–1700 for 50 h.

Microwave gel combustion route is a very simple and scalable process for nanopowder synthesis. In our earlier works upto 65% transmission for Nd:Y_2O_3 was achieved by this process [18,19]. In the present work 1 at% neodymium ion doped Yttrium oxide transparent ceramic with 76% transmission has been achieved with scandium ion addition, from fine, close to spherical nanopowders synthesized by nitrate alanine microwave gel combustion route, on vacuum sintering at 1765 °C for only 40 min.

2. Experimental

Yttrium oxide, Y_2O_3 (99.99% purity Alfa Aesar), Neodymium oxide, Nd_2O_3 (99.999% purity Alfa Aesar) and Scandium

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oxide, Sc_2O_3 were used as the starting materials in the molar ratio of Y:Sc:Nd to be 1.48:0.5:0.02. Sol was prepared by adding oxides, dissolved in nitric acid, and L-Alanine ($\geq 99.5\%$, Fluka) fuel in deionized water. The total cation to fuel molar ratio was 1:0.5. The solution of nitrates and the fuel was combusted in a modified domestic microwave oven operating at 2.45 GHz. Combustion took place within 3–5 min in the microwave oven giving powder precursors. The same series of experiment was carried out without Scandium ion addition. These precursors were calcined at 1000 °C for 2 h.

Powders were milled and uniaxially pressed in a 13 mm diameter steel die lined with tungsten carbide at 34 MPa. The pellets were then pressed isostatically at 300 MPa. The green compacts were sintered at 1765 °C for 40 min under vacuum of 1×10^{-3} Pa, in a furnace with Tungsten and molybdenum mesh heaters at heating and cooling rates of 600 °C/h.

X-Ray Diffraction (XRD) was carried out using Philips X-Ray Diffractometer, PW 3020 in 2θ range from 20 to 80° for characterization of phase purity and particle size. Crystallite size was calculated using Scherrer's equation [20]

$$t = \frac{0.9\lambda}{(\beta^2_{sample} - \beta^2_{inst})^{1/2}\ \cos\ \theta}$$

where t is the crystallite diameter, $\lambda = 1.54056$ Å, θ is the diffraction angle, β_{sample} is the full width at half maximum (FWHM) measured for sample and β_{inst} is FWHM of a silicon standard with large crystallite size (> 150 nm) that was used to determine instrument broadening. Neodymium ion (Nd^{+3}) and scandium ion doping was confirmed by Energy Dispersive X-ray Spectroscopy (EDX) of gold coated compacts of powder sample using scanning electron microscope EVO 40. Particle size range was determined by direct observation technique of Transmission Electron Microscopy (TEM) which measures particle size coupled with observation of particle shape and extent of agglomeration. It was done on FEI Philips Morgagni 268 by preparing samples on copper grids. Transmission of sintered and polished samples was done by Cary 5000 UV–vis–NIR spectrophotometer.

The absorption spectra were measured with Bruker Vertex 70 FTIR spectrophotometer. The emission spectra of specimens were recorded using 808 nm 3 W diode laser as the excitation source and the emission was coupled to a monochromator (Acton SP2300) attached with a InGaAs detector. The spectral measurements were carried out at room temperature within the spectral region of 900–1200 nm with 0.1 nm resolution. The fluorescence lifetime was measured by excitation with 808 nm laser diode and luminescence decay curve at 1084 nm for $Nd_{0.02}Sc_{0.5}Y_{1.48}O_3$ was recorded.

3. Result and discussion

From XRD patterns (Fig. 1) of $Nd_{0.02}Sc_{0.5}Y_{1.48}O_3$ and $Nd_{0.02}Y_{1.98}O_3$ nanopowders all peaks correspond to that of cubic Yttrium oxide are seen and no other phases were detected. In addition, owing to the discrepancy of ionic radii between Sc^{3+} and Y^{3+} ($R_{Sc} = 74.5$ pm, $R_Y = 90.0$ pm [21]), the substitution of Y^{3+} sites by Sc^{3+} ions will cause a certain degree of lattice distortion. As a result, the diffraction peaks of $Nd_{0.02}Sc_{0.5}Y_{1.48}O_3$ shift to higher angles as compared with those of $Nd_{0.02}Y_{1.98}O_3$. The lattice constant for $Nd_{0.02}Sc_{0.5}Y_{1.48}O_3$ calculated by the least square method was found to be 10.511 Å (for undoped Y2O3 $a = 10.604$ Å from JCPDS 88-2162) [22] indicating substitution of Y^{+3} ions by Sc^{+3} ions. XRD of post sintered $Nd_{0.02}Sc_{0.5}Y_{1.48}O_3$ ceramic (Fig. 2) showed all the peaks characteristic of cubic Yttrium oxide with shift of diffraction peaks to higher angles, indicating retention of phase purity during sintering.

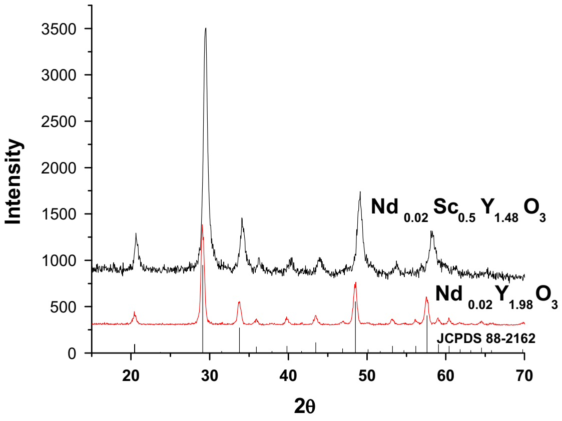

Fig. 1. XRD of nanopowders calcined at 1000 °C for 2 h.

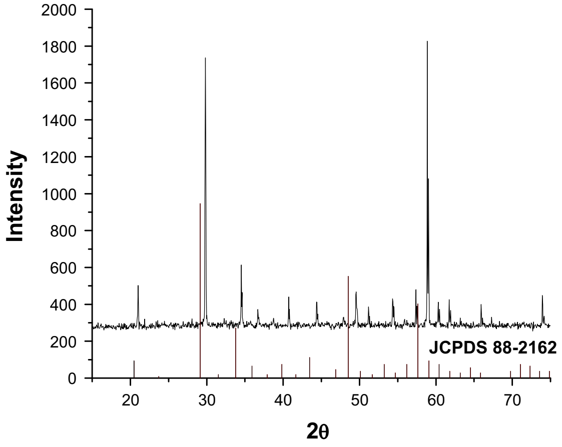

Fig. 2. XRD of Nd $_{0.02}$ $Sc_{0.5}$ $Y_{1.48}$ O_3 sintered ceramic.

Powder EDX spectra (Fig. 3) confirmed the doping of Nd^{3+} and Sc^{+3} ions. Peaks of all constituent elements Y, Sc, Nd and O are observed in the EDX spectrum of Nd:Y_2O_3 nanopowders.

For both $Nd_{0.02}Sc_{0.5}Y_{1.48}O_3$ and $Nd_{0.02}Y_{1.98}O_3$ nanopowders, morphology as observed by TEM (Fig. 4) shows particles in the size range of 25–40 nm, with uniform morphology close to spherical, thus are expected to undergo fast densification during sintering [23]. However $Nd_{0.02}Sc_{0.5}Y_{1.48}O_3$ undergoes rapid densification at 1740 °C for 40 min compared to very less densification in case of $Nd_{0.02}Y_{1.98}O_3$. This is due to the presence of scandium ion, which form solid solution with yttrium oxide and prevents grain growth and leads to rapid sintering.

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