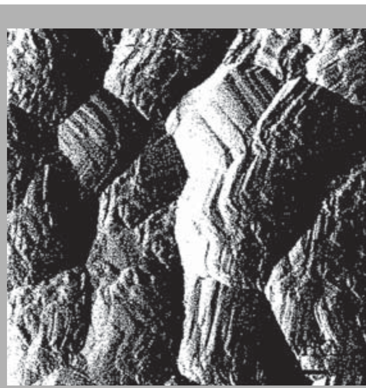
Abstract: Micro-hardness and fracture toughness, as well as linear optical properties (full transmission spectrum and refractive index dispersion) of fine-grained Lu_2O_3 ceramics fabricated by VSN method are presented.



AFM image of Lu_2O_3 ceramics fabricated by VSN method

Mechanical and optical properties of Lu_2O_3 host-ceramics for Ln^{3+} lasants

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1. Introduction

The optical technology using the $\chi^{(3)}$-nonlinear Kerr lens mode locking (KLM) effect (see, e.g. [1]) has been applied quite recently to generate ultra-short pulses in solid-state lasers based on novel gain materials – highly transparent ceramics with lanthanide lasants (Ln^{3+}). So, femtosecond pulses with a duration of 70 fs and of 65 fs, respectively, were obtained in KLM lasers, based on fine-grained Sc_2O_3 and Lu_2O_3 ceramics doped with Yb^{3+} [2]. Quite recently, with the help of the long-known idea of *combined active medium* (CAM) [3] also the record of short pulses for solid-state crystalline Ln^{3+}-lasers were achieved. In this experiment, with the combination of Y_2O_3+Sc_2O_3 ceramics, a more broad effective Yb^{3+}-gain contour (more broad than for each of these ytterbium-doped ceramics individually [4]) allowed to obtain 56 fs pulses (with peak power of about 50 kW per pulse) for this first CAM ceramic laser.

Of course, these values are not a limit, the pulse duration can be decreased further through the optimization of the laser cavity and the selection of ceramic combinations. To achieve much shorter laser pulses, CAM can be not only crystalline (single crystals or ceramics) materials with the same activator lasant, as it is shown in [3] (Yb^{3+}-ion doped Y_2O_3 and Sc_2O_3 ceramics), but can be also identical or different materials doped with different kind of Ln^{3+} activators, e.g. Yb^{3+} and Nd^{3+}, which offer suitable spectral gain contours of their "one-micron" intermanifold

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Ceramics	Optical transparency, μm [a]	n [b]	n_2 [c], 10^{-13} esu	Ln^{3+} lasant	Tunable lasing range [d], μm	Laser pulse duration, fs [e]	ω_{SRS} [f], cm^{-1}	g_{ssR}^{St1} [g], $cm\,GW^{-1}$	H [h], GPa	K_{1C} [h], $MPa\,m^{\frac{1}{2}}$
Sc_2O_3	≈0.23 – ≈6.7 [7]	≈1.96 [7]	≈5.32	Yb^{3+}	1.0405 – 1.0426; 1.0905 – 1.0965	≈70 [2]	≈419	≈0.72 [8]	-	-
Y_2O_3	≈0.23 – ≈8.5 [9]	≈1.89 [10]	≈5.79	Nd^{3+}, Yb^{3+}	1.0333 – 1.0444; 1.0628 – 1.0794	≈188 [11]	≈378	≈0.4 [14]	10	2.5
Lu_2O_3	≈**0.23** – ≈7.7	≈**1.91**	≈3.96	Nd^{3+}, Yb^{3+}	1.0338 – 1.0385; 1.0673 – 1.0813	≈65 [2]	≈392	≈0.3 [15]	**12.5**	**4.1**
Y_2O_3+ +Sc_2O_3	-	-	-	Yb^{3+}	-	≈56 [2] [i]	-	-	-	-

[a] For ≈ 1.5 mm thick polished plates. The UV borders correspond to zero-level transmission, the mid-IR borders are estimated at 50% of maximum transmission.
[b] For a central wavelength (≈ 1.06 μm) of Yb^{3+} generation ($^2F_{5/2} \rightarrow ^2F_{7/2}$ channel) in sesquioxide RE_2O_3 ceramics.
[c] Nonlinear refractive index was measured in [12] by the Z-scan method at a wavelength of 0.532 μm.
[d] Ytterbium two-band CW generation with LD pumping was achieved in [4, 7, 13].
[e] In [2] femtosecond pulses were achieved by KLM method and in [11] by use of a semiconductor saturable absorber mirror (SESAM).
[f] ω_{SRS}: the energy of SRS-promoting vibration mode [8, 14, 15].
[g] The first Stokes steady-state Raman gain coefficient was estimated under excitation by picosecond pulses of Nd^{3+}:$Y_3Al_5O_{12}$ one-micron laser ($^4F_{3/2} \rightarrow ^4I_{11/2}$ generation channel).
[h] Grain size is 1–2 μm. Data of Y_2O_3 ceramics from [16].
[i] The recorded short 56 fs pulses for solid-state Ln^{3+}-lasers were achieved with this combined-active ceramic medium. Before, the best result (61 fs [17]) for crystalline femtosecond lasers had been obtained with a YVO_4:Yb^{3+} single crystal by the use KLM method as well.

Table 1 Mechanical, optical, nonlinear optical and laser data (at room temperature) of fine-grained RE_2O_3 VSN ceramic hosts for Ln^{3+} lasants

transitions ($^2F_{5/2} \rightarrow ^2F_{7/2}$ and $^4F_{3/2} \rightarrow ^4I_{11/2}$ [5]). However, without comprehensive data on physical properties of the rare earth oxide ceramics a materials optimization of these CAM is difficult. Therefore, considering particularly the rare earth oxide Lu_2O_3, for further developments of all types of Lu_2O_3:Ln^{3+} ceramic lasers, as well as for their promotion to applied fields, the detailed knowledge of mechanical and linear optical properties of this novel nanocrystalline material plays an essential role.

This letter reports on the results of the investigation of main mechanical characteristics such as micro-hardness (H) and brittleness (K_{1C}), as well as on the data of precise measurement of the refractive index and its dispersion in wide spectral range of undoped Lu_2O_3 ceramics fabricated by the vacuum sintering and nanotechnology (VSN) method [6]. Properties of this ceramic related directly to Kerr ($\chi^{(3)}$) nonlinearity as nonlinear refractive index n_2 and stimulated Raman scattering (SRS) were investigated earlier (see Table 1). Table 1 is intended also to be a short database on some known laser and nonlinear laser characteristics of VSN sesquioxide RE_2O_3:Ln^{3+} ceramics (RE = Sc, Y, and Lu).

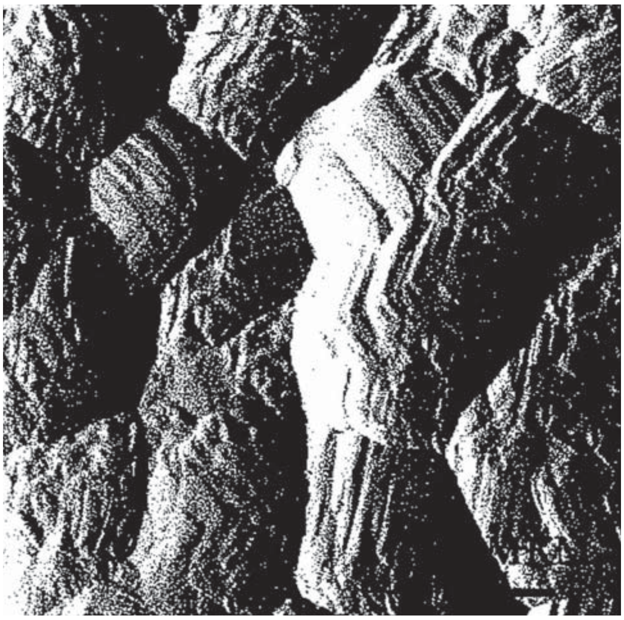

Figure 1 AFM image of Lu_2O_3 ceramics fabricated by VSN method

2. Micro-hardness and fracture toughness

Mechanical properties of crystalline materials are usually studied by the indentation and sclerometric (scratching) methods. These techniques allow to estimate their strength characteristics – hardness (H) and brittleness. The latter characteristics is related to the fracture toughness (K_{1C}) and can reliably be determined from the measured linear dimensions of radial cracks (C) produced by an indenter as a function of the applied load. In our measurements of micro-hardness and fracture toughness Lu_2O_3 ceramic plates with polished surfaces (to the 14-th class of surface finish) were used. The experiments were carried out using a micro-hardness gauge (PMT-3 type) with a diamond Vickers indenter. The micro-hardness was estimated using the well-known formula H = $k(P/d^2)$, where

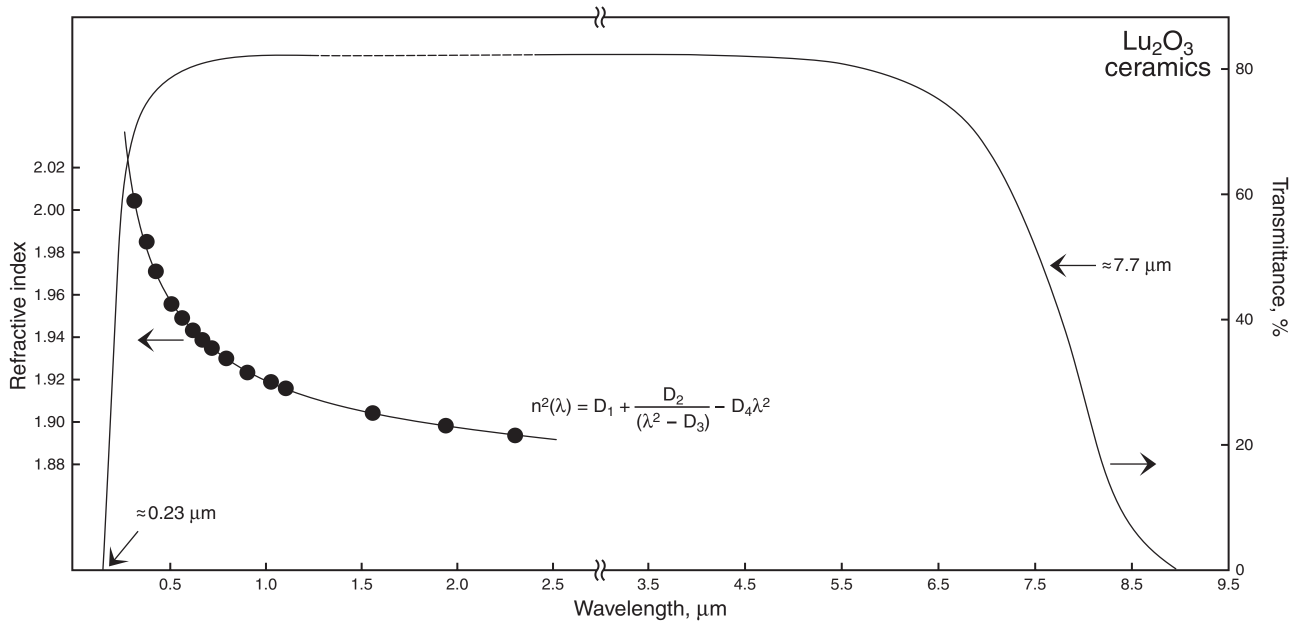

Figure 2 Room-temperature transmission spectrum and dispersion of refractive index of Lu_2O_3 ceramic host for Ln^{3+} lasants. Dots represent measured data, corrected with respect to the refractive index of air (see text), the line gives the Sellmeier fit (see text). The Sellmeier coefficients are: D_1 = 3.61968(9), D_2 = 0.04131(4), D_3 = 0.0238(1), and D_4 = 0.00856(2) (sum of the squares of the residuals of the fit is 5.0×10^{-10})

P is the load applied to the indenter; k is the coefficient dependent on the indenter shape (equal to 1.854 in our case); and d is the diagonal of the indentation print. The fracture toughness is related to P and C values as K_{1C} = $0.016(E/H)^{1/2}(P/C)^{3/2}$ [18] (here E is the Young's module, in our calculation we used E = 178.3 GPa known for polycrystalline Lu_2O_3). The load applied to the Vickers indenter was chosen as to provide indentation prints and radial cracks that could be measured ($P \approx 1N$). The ceramics were fractured as follows: a scratch (stress concentrator) of necessary length was made on the sample surface before the sample was subjected to three-point bending. The morphology of fractured surfaces was investigated by atomic force microscopy (AFM). For the studied Lu_2O_3 ceramics with grain sizes of 1 – 2 μm a layered structure of all grains was found (Fig. 1). The thickness of all layers is approximately the same and equals about 100 nm. An analysis of the deformation structure and morphology of the studied ceramics shows that the layered structure of the grains are attributed to the twinning process on the (111) plane in the [112] direction and the main fracture mechanism is the inter-granular type. Results of conducted experiments are given in Table 1.

3. Linear optical properties

Fig. 2 presents transmission spectra of a polished Lu_2O_3 ceramic plate of ≈ 1.5 mm thickness. In the short wavelength range (0.2 – 1.05 μm) a grating spectrophotometer Hitachi U4100 was used, in the long wavelength range (2.5 – 10 μm) the spectrum was determined by a Perkin Elmer "Spectrum one FT IR" equipment.

The dispersion of the refractive index of Lu_2O_3 ceramics was measured by the prism method with normal incidence on one of the prism faces at 14 discrete wavelengths in the spectral range 0.365 – 2.325 μm using a high precision goniometer-spectrometer system [19]. All data were measured twice with a maximum deviation of the values of 2×10^{-5}. A correction of refractive indices, taking into account the refractive indices of air and its dispersion ($n^{air}(\lambda)$ [20]), is necessary for determination of refractive indices with error less than 1×10^{-4}, all results given here correspond to corrected refractive indices. The corrected experimental data were fitted with a modified Sellmeier equation of the type

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

with the obtained coefficients D_1 = 3.61968(9), D_2 = 0.04131(4), D_3 = 0.0238(1), and D_4 = 0.00856(2). The sum of the squares of the residuals of the fit is 5.0×10^{-10}.

4. Summary

Main mechanical properties of fine-grained VSN host ceramics Lu_2O_3 for Ln^{3+} lasants were investigated. Values of micro-hardness and fracture toughness were determined as H =12.5 GPa and K_{C1} = 4.1 MPa $m^{1/2}$, respectively. A full optical transmission spectrum and the refractive index and its dispersion (in the spectral region 0.365 – 2.325 μm) of undoped Lu_2O_3 ceramics were measured as well.

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