Structural and Spectroscopic Properties of ND Doped YAG Microsize Single Crystals

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Abstract— Micro size single crystals of doped YAG were obtained regulated cooling of supersaturated glass solutions. In this work we investigate Raman and optical spectroscopic properties of these micro-crystals. Most of the measurements were obtaining selecting a single micro-crystal using microscope. All results indicated nearly identical properties compared to a reference bulk crystal grown by the Czochralski method.

Keywords— Nd-YAG micro-crystal, Optical spectroscopy

I. Introduction

In a recent publication, we reported a new methodology capable of synthesizing micro-metric single-crystal garnets containing rare earth ions on a large scale using heavy metal glass as a reaction medium [1]. In this methodology, a glass containing GeO_2, Bi_2O_3, PbO, Ga_2O_3 (and/or Al_2O_3 and/or Fe_2O_3), in addition to the desired rare earth, is melted at ~1200°C. During the cooling process, micrometer-size cubic crystals precipitate and grow from the supercooled liquid. In this work we report structural and spectroscopic properties of these Nd:YAG microcrystals compared to a Nd:YAG bulk crystal, grown by Czochralski method. Other samples (in the form of bulk crystals and crystalline ceramics) with different Nd^{3+} concentration were also considered in the study for reference in the spectroscopic measurements.

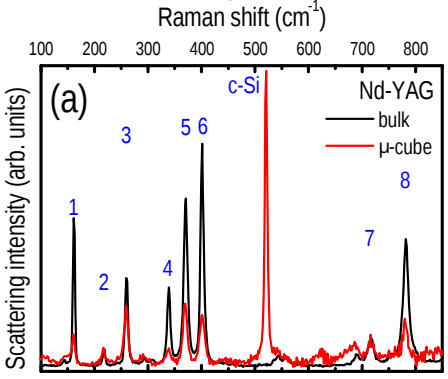
II. Results

Further details regarding the production of the Nd:YAG μ-cubes can be found in ref. [1,2]. All optical measurements were performed at room-temperature and followed the same experimental conditions.

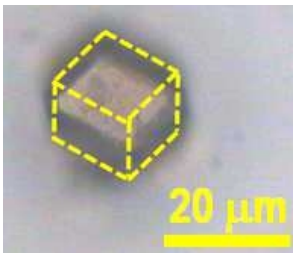
The Raman measurements (non-polarized and resulting from back-scattering geometry) employed an optical microscope and 632.8 nm laser radiation as described in [3]. A spot size of typically 1 μm^2 was adopted during the measurements and great care was taken to avoid accidental sample heating. Figure 1 shows the results obtained from a single Nd:YAG micro-cube: (a) the Raman spectra of a μ-cube and of a reference (bulk) 0.4% doped Nd:YAG grown by the Czochralski method, (b) the optical micrograph of the considered μ-cube, and (c) its corresponding Raman image, as obtained from the scattering signal at ~ 400 cm^{-1}. In Fig. 1(a) the signal at 520 cm^{-1} is due to the crystalline Si wafer, over which the μ-cubes are positioned. The observed Raman spectra are in excellent agreement with previous data observed in Nd:YAG single crystals [4].

The fluorescence spectrum was also obtained in a microscope from a singleμ-crystal under 632.8nm excitation. All NIR characteristic Stark lines of Nd:YAG, emissions from $^4F_{5/2}$, $^2H_{9/2}$ and $^4F_{3/2}$ levels, were observed [5,6]. The spectra the μ-crystal was compared with the reference bulk crystal and no difference was observed. Figure 2.(a) shows the partial Nd:YAG energy level diagram with the main NIR emission lines: $^2H_{9/2}$,$^2F_{5/2} \rightarrow {}^4I_{11/2}$ (A), $^4F_{3/2} \rightarrow {}^4I_{11/2}$ (B) and $^4F_{3/2} \rightarrow {}^4I_{9/2}$(C). Figure 2.(b) shows a comparison of the B lines obtained in a bulk crystal and a single μ-crystal microcrystal.

Figure 3 shows the optical absorption spectrum (as obtained from reflectance measurements) of a mixture consisting of single Nd-YAG μ-cubes and the residuals of



(a)



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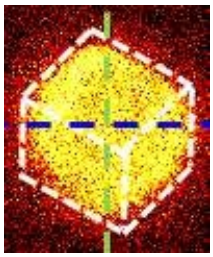
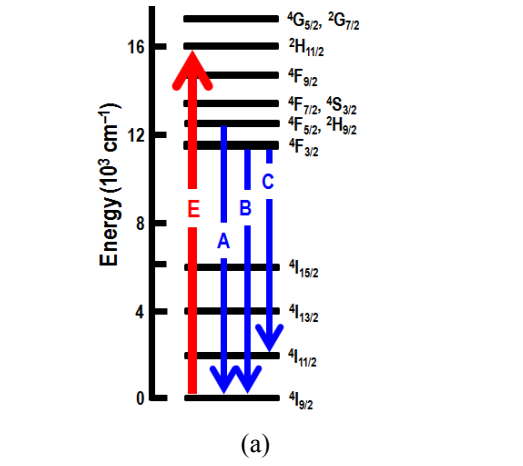


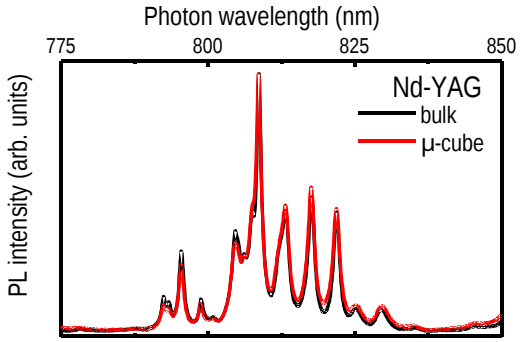
(c)

Fig. 1– Experimental results obtained from a single Nd-YAG μ-cube: (a) Raman scattering spectrum in aμ-cube compared to a reference bulk crystal; (b) optical micrograph (500x magnification) and (c) Raman imaging

(scattering signal at 400 cm^{-1}), making evident the typical shape and homogeneity of the μ-cube. The dashed lines in (b) and in (c) are just guides to the eye.



(a)



(b)

Fig. 2–(a) Nd $^{3+}$:YAG energy level diagram. Under 808 nm excitation (E), the main emission lines are indicated by A, B and C (b) optical emission spectra (at room-temperature and with 632.8nm photon excitation), in the 775–850 nm range (A), as obtained from a bulk Nd:YAG crystal and from a single Nd:YAG μ-cube.

their former glass matrix. According to the spectrum, the effect of the glass residuals is evident by the increasing absorption at shorter wavelengths. For comparison reasons, Fig. 3 also shows the optical absorption spectrum of the Nd:YAG bulk sample.

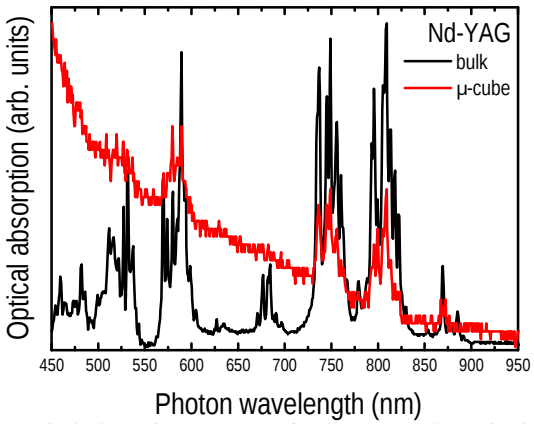


Fig. 3– Optical absorption spectrum of Nd:YAG μ-cube (mixed with the glass remains) and of bulk Nd:YAG reference (bulk) crystal.

concentration due to a cross-relaxation process leading to a decrease in the fluorescence quantum efficiency. In fact, the value τ ~133 μs is expected for a crystal or microcrystalline ceramic with Nd concentration ~3 at.%. The longer lifetime (τ ~ 217 μs) shown in Fig.4(b) indicates a Nd concentration ~ 1 at.%. Therefore, the fluorescence decay data suggests that the μ-cubes grown by this processes present a Nd^{3+} concentration smaller than the concentration of the precursor glass-ceramic. In fact, this hypothesis was corroborated by x-ray spectroscopic data.

III. CONCLUDIND REMARKS

In spite of their very small dimensions (typically in the 10–15 μm range) the present study confirms the presence of optically active Nd^{3+} ions in the μ-cubes. Likewise, the Raman results confirm on the effect of different Nd concentrations as well as eventual practical applications (random laser, temperature probes, etc.).

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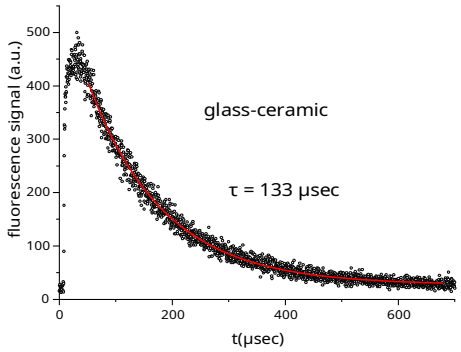
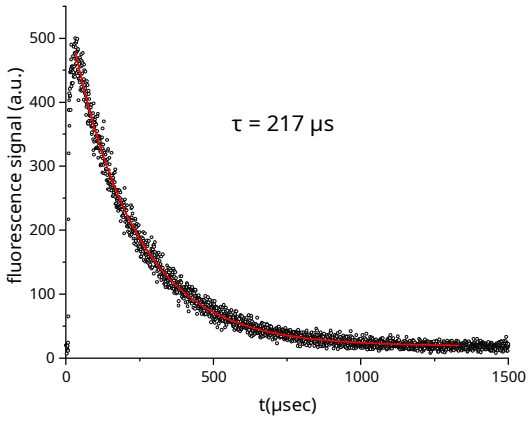


(a)



(b)

Fig. 4– Fluorescence decay time due to Nd^{3+} ions, as obtained from a glass-ceramic sample (a) and from the μ-cubes (b). The measurements were made at room-temperature by exciting the samples with 808 nm photons. The fit curves indicate a good agreement with a single exponential behavior.

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