Sixth quarterly report on the grant N_0 N00014-96-1-5015 "Powder laser development and demonstration".
(Covering the period 04/15/97-07/15/97)

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The effects of the pumping light penetration and the powder density to the stimulated emission in powders of solid-state luminophosphors

Abstract

A laser-like emission without a cavity was obtained (at $\lambda \approx 800$ nm) in powders of Ti-sapphire laser crystal and compared to that in $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$. The effect of the material volume density on the stimulated emission in $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ was experimentally studied. We have found that formation of a hole in a powder sample by a pumping laser beam is advantageous for stimulated emission in Ti-sapphire and disadvantageous in $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$. Using the results of our experimental studies of light propagation in powder laser media, we evaluated the penetration depth and the absorption efficiency in the materials studied and explained most of experimental laser results.

1. Introduction

A laser-like emission in scattering media without a cavity has been observed in powders of solid-state luminophosphors [1-6] and dye solutions with scatterers [7-9]. This effect is characterized by a narrowing of the emission line and generation of short pulses of light, at the pumping energy exceeding some critical threshold value. In solid-state materials, the wavelength tuning in a narrow spectral range was demonstrated in Nd doped powder lasers in Refs. [3,6].

Almost all studies in solid-state powder lasers described in the literature were done using Nd doped materials. Recently, a stimulated emission was observed in pulverized LiF with color centers [10]. In conventional solid-state lasers, the widest spectral tuning range and the shortest linewidth-confined emission pulses (see for example Ref. [11]) are obtained in a Ti-sapphire laser first demonstrated by Moulton in Ref. [13]. In the present work, we obtain stimulated emission in the powder of Ti-sapphire laser crystal and compare it to stimulated emission in scattering Nd doped materials.

According to Refs. [3,6], a laser-like emission in powders of Nd doped luminophosphors is determined by a collective behavior of many excited powder particles. In the present work, we study the efficiency of stimulated emission as a function of the volume density of a scattering media.

We also have found that a hole "drilled" in a powder sample by an intense focused pumping beam is advantageous for stimulated emission in Ti-sapphire powders and disadvantageous for that in Nd doped powders. We explain this effect by the different ratio of the scattering penetration length and the effective absorption length in Nd doped and Ti doped materials. To confirm this model, we have studied the propagation and reflection of the

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pumping light in powders of different crystals experimentally. Using a simple two-flux theory for the wave propagation in a scattering media, we estimated the coefficient of scattering, the penetration depth and the absorbed energy in various powders, and explained some of the stimulated emission experimental results.

2. Materials studied

In the stimulated emission experiments and the light propagation experiments, we used the following materials:
- powders and ceramics of sintered $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$;
- powders of Ti-sapphire laser crystal, with Ti concentration equal to 0.2% and 0.1%;
- $1\mu m$ Al_2O_3 polishing powder from Buehler, Ltd.

The powder and ceramic samples of $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ were synthesized by a solid-state reaction of mixed and pressed starting components. The powders of Ti-sapphire laser crystals were prepared by grinding Czochralski grown laser crystals. The average particle size in Nd doped powder was ≈ 3.5 μm and the average size of particles in Ti-sapphire powders was ≈ 3 μm.

An absorption spectrum of $Nd_{0.5}La_{0.5}Al_3(BO_3)$ consists of a large number of lines ranging from ultraviolet to infrared. Averaged over different polarizations, the absorption coefficients in bulk $Nd_{0.5}La_{0.5}Al_3(BO_3)$ at several particular wavelengths of interest, k_{abs}, are presented in Table 1 [13]. At weak excitation, the luminescence decay-time in (scattering) $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ is 18 μs; the multi-line $^4F_{3/2} \rightarrow ^4I_{11/2}$ luminescence spectrum has a maximum at 1063.1 nm.

The broadband absorption in Ti-sapphire is centered at ≈ 460 nm and is characterized by a full width at half height (FWHH) approximatelly equal to 110 nm. The absorption coefficients (k_{abs}) in 0.2% doped Ti-sapphire are listed in Table 1. The luminescence in Ti-sapphire is characterized by a very wide, FWHH≈ 230nm, spectral band centered at ≈ 800nm. The room temperature decay-time of Ti-sapphire luminescence is equal to 3.2 ms[*].

Al_2O_3 is a nominally transparent (nonabsorptive) material.

3. Stimulated emission. The effects of the powder density and pumped volume geometry

In the stimulated emission experiments, powders were placed in a 10 mm thick cuvette and pumped with 15 ns pulses of the frequency doubled Nd:YAG laser.

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[*] - Some spectroscopic parameters of Ti-sapphire are available in Ref. [12]. The other data were kindly provided by Union Carbide Corporation, where the crystals were grown, and obtained in our experiments.

To study the effect of the volume density of a scattering lasing material on the stimulated emission, we performed experiments with low density powders, tightly compressed powders, and a ceramic of $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$. The input/output curves for the stimulated emission registered at 1063.1 nm are presented in Figure 1a. As follows from Figure 1a, the samples with higher volume density are characterized by higher slope efficiency and lower threshold than the low density samples.

Since in Nd doped luminophosphors the overall stimulated emission was stronger for samples with the higher volume density, we began our Ti-sapphire laser experiments with strongly compressed powders. (We define the volume density as the ratio of the density of a powder or ceramic to the density of a single crystal). However, in high density Ti-sapphire powders, we could not obtain stimulated emission below the damage threshold for the cuvette. To our surprise, laser-like emission, characterized by short pulses and substantial narrowing of the emission spectrum with the maximum at $\approx$800 nm, was relatively easily obtained in a low density sample, where a focused pumping beam "drilled" a narrow hole in the powder (an effect similar to the effect of ablation). The diameter of the hole was of the order of 1 mm at the front surface of the cuvette and strongly reduced, down to the 100 - 300 μm range, at the depth of 3 - 5 mm (Figure 2).

According to Figure 3a, showing stimulated emission in the Ti-sapphire powder in response to n_1^{th}, n_2^{th}, n_3^{th} ($n_3 > n_2 > n_1$) pumping pulses, which continuously increased the hole in the powder, the formation of a hole was advantageous for stimulated emission in this material. The experiments with Nd-doped powders demonstrated an opposite behavior: the creation of a hole in the powder caused the reduction of the stimulated emission efficiency, Figure 3b.

The negative effect of the hole formation to the stimulated emission in Nd-doped materials can also be seen in Figure 1a, where the saturation of the slope efficiency in a low density sample is related to the creation of a small hole in the powder volume by a pumping beam.

We believe that the difference in the behavior of Nd doped and Ti doped powders is related to different penetration and absorption of pumping light in those materials. In low absorbing scattering materials, a large portion of the incident pumping energy is not absorbed in the volume but diffusely reflected (scattered) off the sample. A creation of a hole in the material with weak absorption and strong scattering can be advantageous for the stimulated emission, because the hole geometry (first approximation to the "black body") helps to reabsorb pumping photons, otherwise scattered off the powder sample, and increase the net absorbed pumping energy. In the opposite case, in the material with strong absorption and weak scattering, a creation of a hole will lead to redistribution of practically the same (large) amount of the

absorbed pumping energy over a greater volume surrounding a deep and narrow hole. This situation is disadvantageous for the stimulated emission.

4. Reflection and transmission of light in powders of laser crystals

4.1. Model

In the powders of solid-state materials, light propagates in a highly irregular manner, both inside and outside the particles. The boundaries of large particles, small particles, as well as small air gaps between particles play the role of scatterers. In the present work, we use the most general two flux theory [14] to describe the propagation of a plane wave in a scattering medium. This theory does not require any additional assumptions on the nature of the scatterers, and based on the model considering two light fluxes F_+ and F_- traveling in opposite (inbound, z, and outbound -z) directions, with the energy exchange between the fluxes due to scattering.

According to Ref. [14], the propagation of the two fluxes in a scattering medium is described as

$$F_+(z)=C_1 e^{\alpha z}+C_2 e^{-\alpha z},$$ (1)
$$F_-(z)=C_1 A^{-1} e^{\alpha z}+C_2 A e^{-\alpha z} \quad ,$$

where α is the inverse effective penetration depth given by $\alpha = (K(2K+S))^{1/2}$; S and K are correspondingly the effective flux scattering and the effective flux absorption coefficients in the medium; $A=S/(S+K+\alpha)$; and C_1 and C_2 are the coefficients determined by the boundary conditions. According to Ref. [14], the coefficient K describes the absorption of the flux and is equal approximately to $2k_{abs}^{eff}$, where k_{abs}^{eff} is the effective absorption coefficient for the plane wave in the scattering medium. For a powder sample of thickness z_0, the coefficients C_1 and C_2 can be found as [14]

$$C_1 = (1 - r_i)(A - r_e)\exp(-\alpha z_0)/\Delta ,$$
$$C_2 = (1 - r_i)(r_e - A^{-1})\exp(\alpha z_0)/\Delta , \text{ and} $$ (2)
$$\Delta = -(A - r_e)^2 \frac{\exp(-\alpha z_0)}{A} - (1 - r_e A)^2 \frac{\exp(\alpha z_0)}{A} ,$$

where r_i and r_e are the effective Fresnel coefficients describing reflection for inbound and outbound light at the material/air boundary.

For a semi-infinite medium, the reflectance, R, can be found as

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$$R = r_i + \frac{(1-r_e)(1-r_i)A}{(1-r_e A)}, \tag{3}$$

The transmittance, T, of a thin scattering sample of the thickness z_0 is given by [14]

$$T = (1-r_i)F_+(z_0), \tag{4}$$

where $F_+(z)$ is determined by Equations (1) and (2). $T(z_0)$ depends on the sample thickness, z_0, approximately as $\exp(-\alpha z_0)$ [14].

4.2. Experimental measurements of reflection

Experimentally, we registered the intensity, R, of the pumping light (at 532 nm) scattered off a thick cuvette containing powder or a piece of ceramic. The scattered light was registered in a small solid angle, the same for all measurements, that did not coincide either with the direction of exact backscattering or the direction of the beam reflected by the cell wall. The experimental data on the reflectance off different materials doped with different ions in different concentrations are summarized in Table 2.

The experimental data presented in Table 2, are normalized to reflectance off the Al_2O_3 powder (the material without absorption), which is taken to be equal to unity. All powders used in this particular experiment, were medium-dense. The dependence of the reflectance on the volume density in Nd doped luminophosphors is shown in Table 3.

As follows from Tables 2,3, there is a strong difference between absorption in Nd and Ti doped powders: the former one absorbs approximatelly a half of the incident pumping energy, whereas the absorption in the latter one is only about 0.1.

4.3. Experimental measurements of the transmission.

In this particular measurement, the powders of Ti-sapphire (0.2 %), $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$, and Al_2O_3 materials were filled into cuvettes of different thickness and illuminated with a laser beam, which diameter was comparable or larger than the thickness of the cuvette. Using a frequency doubled Nd:YAG laser (532 nm), an Ar laser (514.5 nm), and a He-Ne laser (632.8 nm), we measured the transmittance of the powder at different wavelengths, where the absorption coefficient was high or low, Table 1. Typical experimental dependencies obtained in the transmission experiment are shown in Fig. 4.

4.4. Evaluation of the scattering and penetration parameters

Comparing the experimental data on reflection and transmission with the theoretically calculated ones, Eqs. (3),(4), we found that the two flux model [14] satisfactorily describes the propagation of light in the scattering laser materials studied. Fitting the experimental data with Eqs. (3),(4), we have evaluated the scattering coefficient, S, and the penetration depth, α^{-1}, for some of our samples. According to Ref. [15], the internal reflection, r_e, in our calculations, was assumed to be equal to 0.7. The effective absorption coefficient in the scattering medium can be recalculated from that in a bulk material using a formula

$$k_{abs}{}^{eff} = k_{abs}\rho, \qquad (5)$$

where ρ is the volume density of the scattering material. Thus,

$$K=2k_{abs}{}^{eff} =2k_{abs}\rho. \qquad (5a)$$

In calculations, we set the medium volume density to 0.6 (the typical number for most of our samples, see Table 3).

The data on transmission in 1 μm Al_2O_3 powder at 632.8 nm can be fit with Formula (4) at $S=5000$ cm^{-1} and $k_{abs,532}=0.01$ cm^{-1} . These values seem to be reasonable for Al_2O_3. In fact, Al_2O_3 is transparent in bulk, and the determined absorption coefficient of Al_2O_3 powder is also very small. The scattering coefficient, S, determined is related to the particle size, d, as $S^{-1}=2d$.

The experimental data on reflection and transmission in Ti(0.2%)-sapphire powder at 532 nm and 514.5 nm can be described satisfactorily with the two-flux model using $S=2000-2500$ cm^{-1} and K corresponding to $k_{abs,532}=2.5$ cm^{-1} and $k_{abs,514.5}=2.7$ cm^{-1} (Formula 5a). The value of the scattering coefficient, S, is related to the particle size $d\approx3$ μm as $S^{-1}\approx1.5d$. The penetration depth for the pumping light (λ=532 nm) in Ti(0.2%)-sapphire powder can be evaluated as 80-100 μm. The experimental data on reflectance in Ti(0.1%)-sapphire powder at λ=532 nm can be satisfactorily fitted by Formula (3), with the same scattering coefficient and two times lower absorption coefficient.

The transmission in $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ powder at 633 nm and 532 nm can be fit relatively well with the two flux model [14] at the coefficient K corresponding to absorption coefficients in bulk $Nd_{0.5}La_{0.5}Al_3(BO_3)$, $k_{abs,532}=12.5$ cm^{-1} and $k_{abs,632.8}=0.8$ cm^{-1}, and $S=2200-2500$ cm $^{-1}$. These numbers correspond to $\approx$40 μm penetration depth at 532 nm and reflectance equal to 72%. The last value is close to that experimentally measured in the low density Nd doped sample, Table 1, but is higher than the reflectance experimentally determined in samples with a higher volume density.

The larger absorbed efficiency experimentally determined in high density samples cannot be explained only by slightly larger absorption coefficients. We suggest that the strong decrease of

reflection in Nd doped powders (at λ=532 nm) with the increase of the volume density can also be ascribed either to the increase of the internal reflection coefficient, r_e, or the decrease of the effective scattering coefficient, S. Further studies are needed to clarify the effect of the powder density on the propagation of pumping and emission in powder lasers.

Thus, we have shown that the propagation of light in scattering laser media can be described in first approximation by a simple two flux model [14], with the effective absorption coefficient related to the absorption coefficient of the bulk material, and effective scattering coefficient approximately equal to half of the inverse particle size.

The penetration depth for the pumping photons was found to be $\approx$40 μm in the $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ powder, and $\approx$100 μm in the Ti(0.2%)-sapphire powder. Experimentally, we have found that the sample of Ti(0.2%)-sapphire powder absorbs about 14% of the incident pumping power while the absorption in $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ powders and ceramic is much higher, 30-50 %, depending on the sample density. The reflection coefficients above are in a reasonably good agreement with those theoretically calculated based on the light penetration experiments.

5. Discussion

The results obtained on reflection and penetration of light support our simple model (introduced in Section 2) explaining why the hole formation is advantageous for stimulated emission in Ti-sapphire powder and is disadvantageous for stimulated emission in Nd doped powder. In fact, the experiments have shown that the absorption in scattering $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ is high. The formation of a hole in $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ does not increase substantially the absorbed pumping energy but leads to the unfortunate for stimulated emission form-factor. In the opposite case, in Ti doped powders, the absorption is low (about 0.1). The formation of a hole (which is a first approximation to the black body) results in a strong increase of absorbed pumping energy and is advantageous for the stimulated emission, in spite of the unfortunate change of the form-factor.

The input/output curves normalized to the absorbed pumping energy for stimulated emission in $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ samples of different volume density are shown in Figure 1b. In conventional lasers, such a behavior of input/output curves (similar slopes and different thresholds) is typical for a series of measurements with different output couplers and the internal loss much less than the smallest output coupling. The analogy with conventional lasers also implies that the effective output coupling is higher in high density scattering samples than in low density samples. As was shown in Ref. [6], in powder lasers, the inverse residence time for the emission photon in a pumped volume, $1/\tau_2$, is a measure of the effective output coupling. Thus, Figure 1b implies that the effective photon residence time is lower in high

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density scattering materials than in low density materials. As was discussed in Section 4.4, there are three potential reasons for higher absorption efficiency in dense scattering media than in low density materials. These are 1) the slightly higher absorption coefficient, 2) the weaker scattering, or 3) the higher internal reflection coefficient, r_e. It is easy to show that only the first of the three reasons above can lead to the decrease of the effective thickness of the pumped layer, the decrease of the emission photon residence time, and, hence, the increase of the effective output coupling.

6. Summary

Stimulated emission without a cavity (at $\lambda \approx 800$ nm) was obtained for the first time from the powder of Ti-sapphire, a high performing, broad band solid-state laser material.

The effect of the pumped volume configuration on stimulated emission, the "hole effect", was observed in powder lasers. The influence of the hole formation on stimulated emission was found to be qualitatively different in Ti doped and Nd doped materials.

The effect of the material volume density on stimulated emission was studied in $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ powders and ceramic. The overall laser efficiency was higher in high density samples. However, the threshold *absorbed* pumping energy was less in low density powders and the slope efficiency related to the *absorbed* pumping energy was independent of the volume density.

Based on our experimental results on transmission, reflection and absorption of light in powders and ceramics, the light propagation in scattering laser medium was described in terms of the two-flux model [14] and the parameters of the light propagation were evaluated. We suggest that the observed "hole effect" is related mostly to the penetration and absorption of the pumping light in a scattering medium.

The effect of the material volume density to the threshold and the stimulated emission efficiency in scattering laser materials was suggested to be related not only to propagation and absorption of the pumping light but also to propagation of the emission light.

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Wavelength, nm	$Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ [*)	Ti(0.2%):sapphire
514.5	12 cm^{-1}	2.7 cm^{-1}
532	12.5 cm^{-1}	2.5 cm^{-1}
632.8	1 cm^{-1}	0.13 cm^{-1}

Table 1. Absorption coefficients, k_{abs}, in $Nd_{0.5}La_{0.5}Al_3(BO_3)$ and Ti(0.2%):sapphire at 514.5 nm, 532 nm, and 632.8 nm. Absorption coefficients in Ti(0.1%):sapphire are two times smaller than those in Ti(0.2%):sapphire.

Material	Reflectance
$Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ ceramic	0.40
$NdAl_3(BO_3)_4$ powder	0.48
$Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ powder	0.52
Ti-sapphire powder (0.2% of Ti)	0.86
Ti-sapphire powder (0.1% of Ti)	0.88
Al_2O_3 powder	1.0

Table 2.   The reflectance of the 532 nm light off different scattering materials.

sample	low dense	medium-dense	high-dense	ceramic
density	0.48	0.54	0.59	0.63
reflectance	0.66	0.52	0.53	0.40

Table 3.   The reflectance of $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ samples of different volume density.

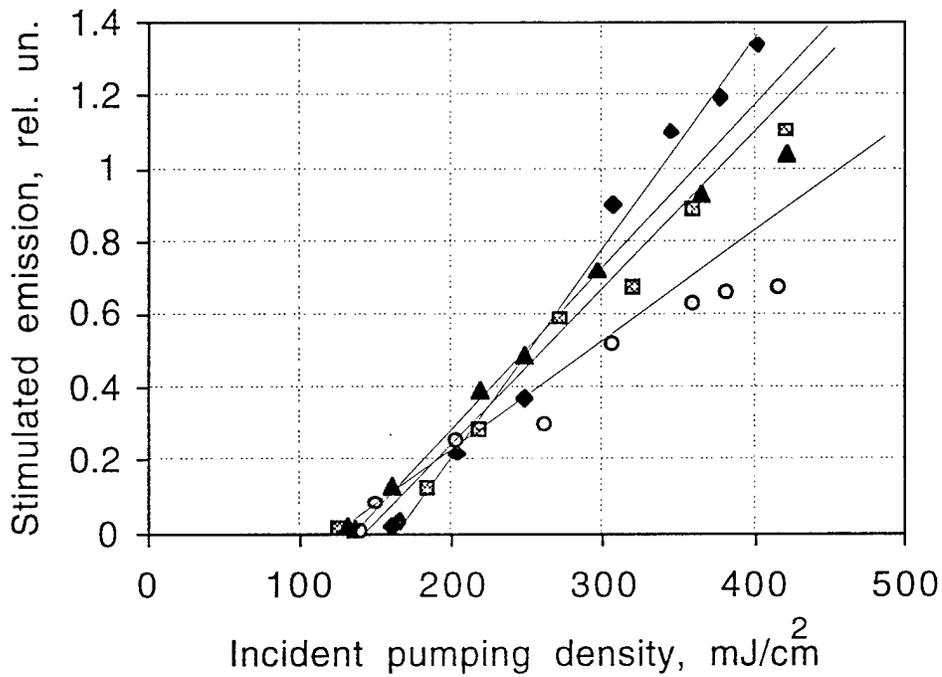[*) - The data in $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ were recalculated from the absorption spectra in published in Ref. [13].

Figure 1a. Stimulated emission *vs* incident pumping energy in $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ powders with the volume density equal to 0.48 (circles), 0.54 (triangles), 0.59 (squares), and in $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ ceramic with volume density equal to 0.63 (diamonds).
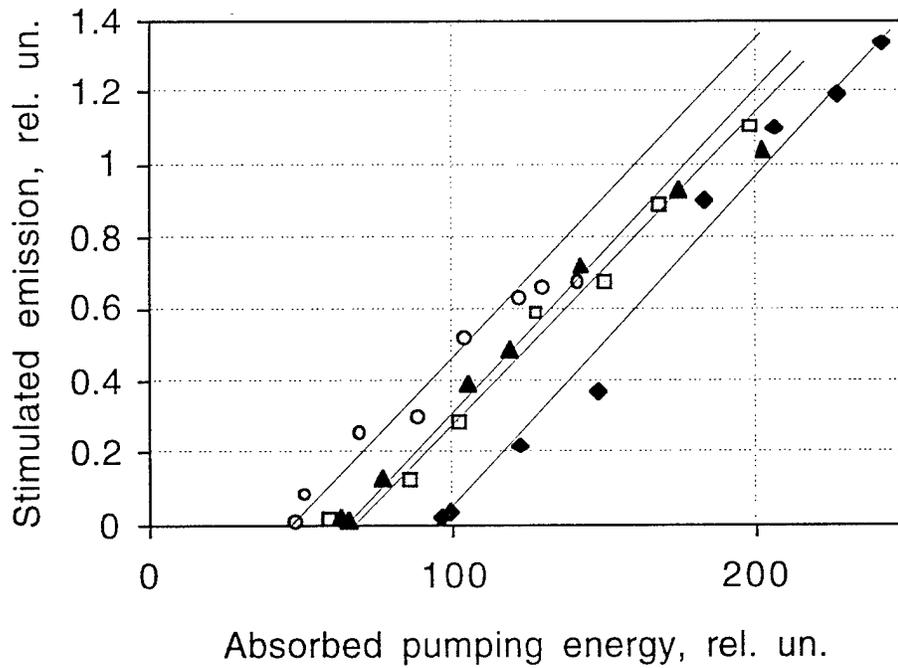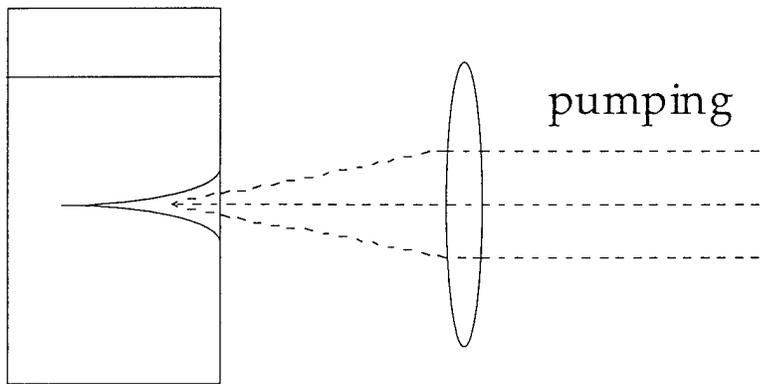

Figure 1b. Stimulated emission *vs* absorbed pumping energy in $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ powders with the volume density equal to 0.48 (circles), 0.54 (triangles), 0.59 (squares), and in $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ ceramic with volume density equal to 0.63 (diamonds).

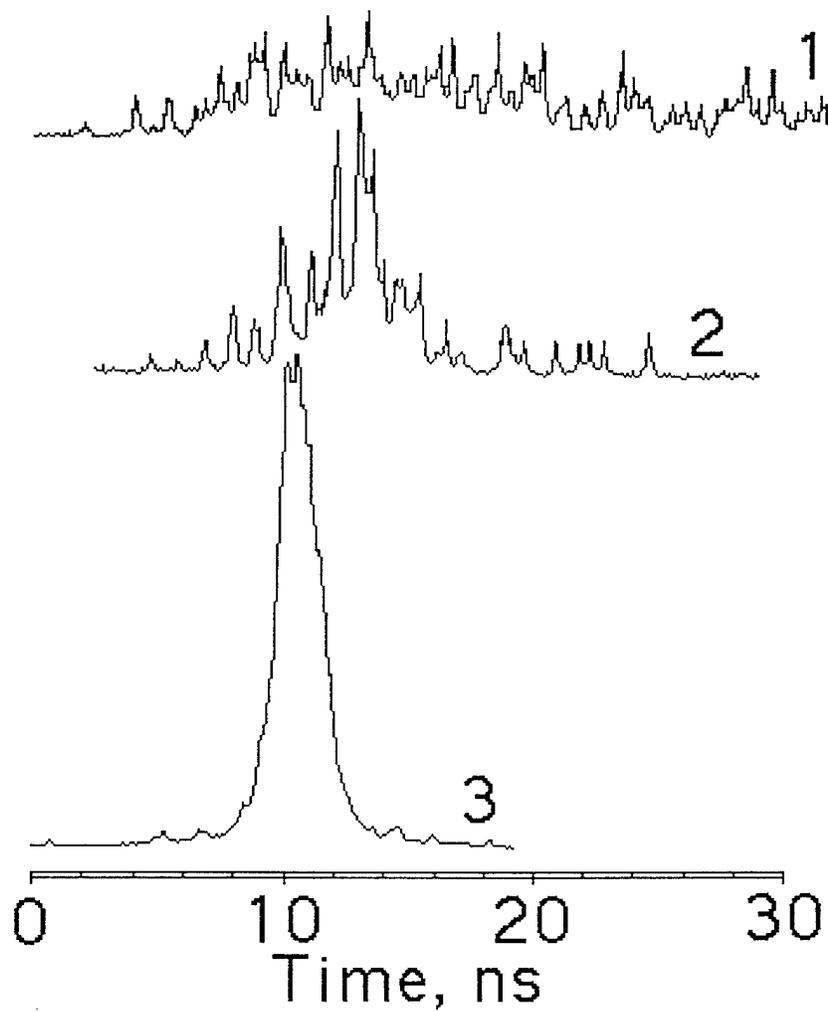Figure 2.   The hole in the powder volume "drilled" by the pumping laser beam.

Figure 3a. Evolution of stimulated emission pulses in 0.2% doped Ti-sapphire powder. Traces 1-3 demonstrate the development of stimulated emission pulses along with the increase of the pumping pulse number and the size of the hole (the intensity scale is different for different traces).
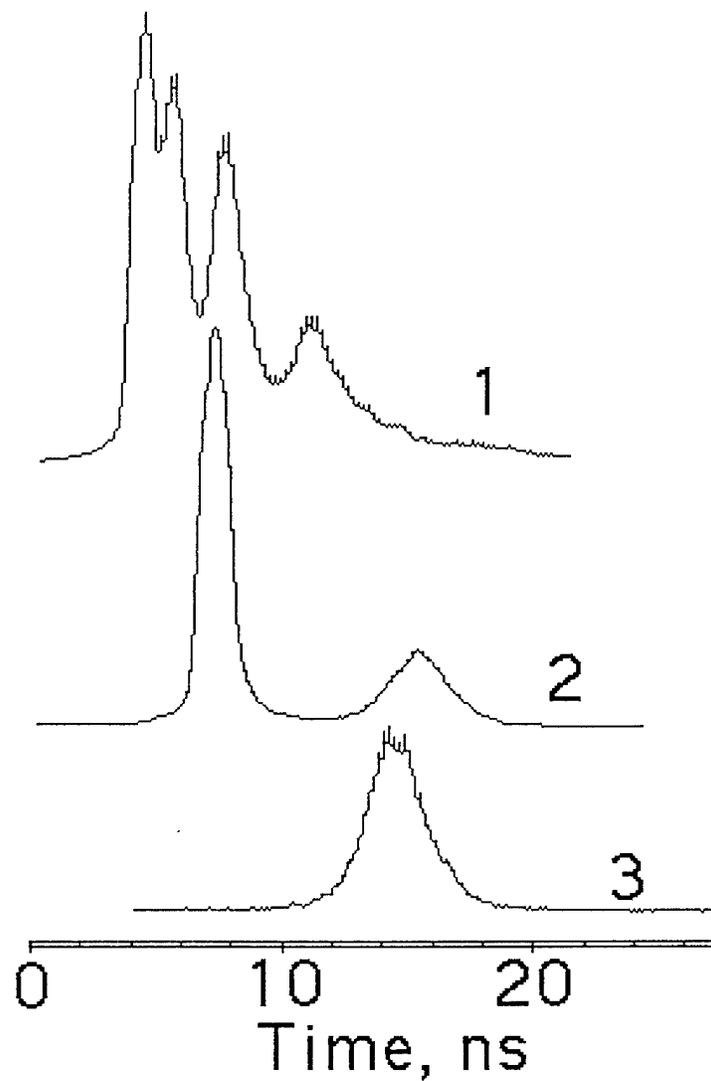
Figure 3b. Evolution of stimulated emission pulses in $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ powder. Traces 1-3 demonstrate the degradation of stimulated emission pulses along with the increase of the pumping pulse number and the size of the hole (the intensity scale is different for different traces).
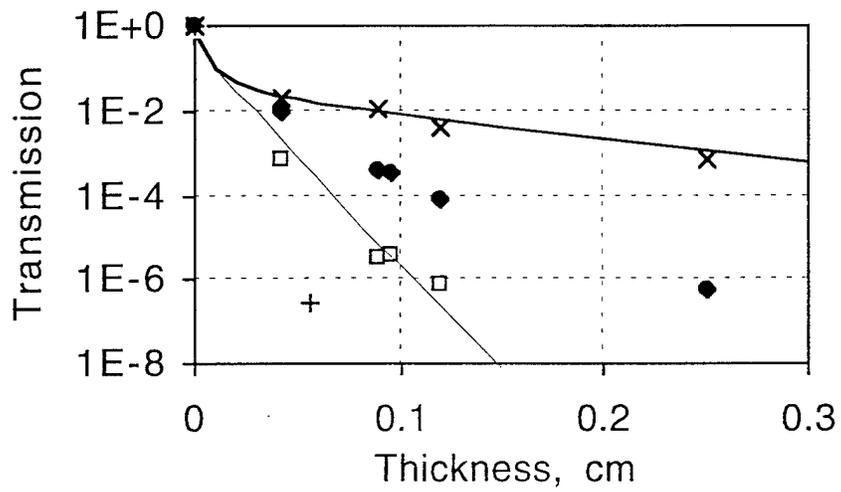
Fig. 4. The transmittance in the dependence of the thickness of the cuvette for Al_2O_3 powder at 632.8 nm (x), $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ powder at 632.8 nm (●), Ti-sapphire powder at 514.5 nm (□), and $Nd_{0.5}La_{0.5}Al_3(BO_3)_4$ powder at 532 nm (+). Solid lines are the theoretical curves calculated according to Equations (1)-(4).