Study of active media based on microparticles of solid-state laser materials

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Abstract. Active media based on particles of solid-state laser materials distributed in liquid or gas may provide a new solution to the problem of heat removal from laser media. We propose active media based on particles of LiF crystal with F₂ color centers, as well as of Nd³⁺-doped crystals and glasses. Mixtures of index-matched liquids with glass and crystal particles, methods of their preparation, and their optical characteristics are considered. The laser characteristics of the active media under excitation by nanosecond laser pulses are compared. A “slurry laser” based on a dense mixture of LiF:F₂ microparticles with an index-matched liquid is demonstrated. Lasing was obtained in small neodymium pentaphosphate crystals (NdP₅O₁₄) and unpolished bulk pieces of concentrated neodymium phosphate glass in immersion liquids.

1. Introduction

Active media based on particles of solid-state laser materials distributed in liquid or gas have been the object of research, since such an approach can provide a new solution to the problem of heat removal during laser operation [1-5]. Glass or crystal nano- and microparticles doped with Nd³⁺ or Yb³⁺ ions were considered as laser media [1-5]. Lasing was obtained in a dispersion of Nd₂O₃ nanoparticles (sizes ≤ 10 nm), scattering losses from such particles were negligible [3, 4]. However, nanoparticles may have some properties (emission lifetime, quantum yield) different from their bulk counterparts, which requires a surface modification of Nd₂O₃ nanoparticles to achieve laser operation [3, 4].

Particles of solid-state laser materials with dimensions over 1 µm are easier to manufacture and need no complicated modification, since their properties are identical to the properties of the original bulk single crystals or glasses. For such particles, index-matched liquids should be used to suppress Fresnel reflection and scattering. A “slurry laser” with an active medium in the form of a dense mixture of particles (sized 200-1000 µm) of LiF crystal with F₂ color centers (CCs) and an immersion liquid was demonstrated in [6]. The possibility of developing such a laser based on particles doped with Nd³⁺ ions has been studied. The “slurry laser” concept may be attractive due to a potentially higher laser output.
energy from a dense pulp circulating through the laser module. In this paper, we present the results of the study of laser media based on particles of LiF:F$_2$, Nd:YLF, and NdP$_2$O$_4$ crystals and Nd:glass. Compositions of immersion mixtures with glass and crystal particles, methods of their preparation, and their optical characteristics are considered; data on excitation of lasing in test samples are presented.

2. Fabrication of index-matched slurry-like active media

Slurry lasers can be based only on high-gain active media, because it is necessary to overcome the internal losses in the mixture of solid particles and a liquid due to incomplete immersion and existence of various scattering centers. LiF crystal with CCs [7-13] is an interesting material for preparing an active medium for slurry lasers. LiF has a high thermal conductivity (4 W·m$^{-1}$·K$^{-1}$) and can be used in cw and pulse-periodic lasers. The refractive indices of irradiated and non-irradiated LiF crystals are almost identical, namely, $n_{LiF} \approx 1.39$. This low value of the refractive index simplifies the search for an immersion liquid for LiF crystal. There are several types of LiF crystals with color centers, i.e., LiF:F$_2^-$, LiF:F$_2^+$, LiF:F$_2$, and some others [7-13]. One of the most promising materials is LiF with F$_2$ CCs. F$_2$ CCs have broad absorption (0.42-0.47 μm) and emission (0.56-0.84 μm) bands, as well as high emission and absorption cross sections, $\sigma \approx 7 \times 10^{-17}$ cm$^2$. Their characteristic feature is a very high concentration of centers, which results in a high amplification gain up to tens of cm$^{-1}$ [7-9].

F$_2$ CCs with a concentration of $10^{18}$ cm$^{-3}$ were produced in nominally pure oxygen-free samples of LiF crystals by irradiation at room temperature with $\gamma$-quanta from a Co$^{60}$ source with a dose of $2 \times 10^{7}$ Rad. The ground LiF:F$_2$ powder was sequentially sieved through sieves with different mesh sizes to separate fractions with sizes from 200 to 1000 μm and larger. Several pieces of transparent non-irradiated LiF crystals were subjected to grinding and fractionation by the same procedure for comparison. Microphotograph of a non-irradiated LiF powder fraction obtained using a mortar with a pestle and then sieved through 200-500 μm sieves is shown in figure 1a. The fractions of crushed material were placed together with an immersion liquid into rectangular glass cuvettes with dimensions of $24 \times 40 \times 8$ mm$^3$ and a 3-mm gap between the walls (figure 1b).

As a rule, it is impossible to find a liquid with a refractive index that precisely coincides with the refractive index of solid particles and has the same dispersion characteristics in a given spectral region. However, it is possible to use a mixture of two liquids with higher and lower refractive indices. In this case, the refractive index of the mixture can be equal to the particle refractive index $n_p$, i.e.,

![Figure 1. Microphotograph of the LiF crystal powder sieved through meshes (a); photo of a 3-mm-thick cuvette containing a slurry of 500-1000 μm yellowish LiF:F$_2$ granules in an immersion liquid (heptane/vaseline oil) and the spot of a He-Ne laser beam passed through this slurry (b).](image-url)
\[ n_p = x \cdot n_1 + (1 - x) \cdot n_2, \]

(1)

Where \( n_1 \) and \( n_2 \) are the refractive indices of the first and second liquids, and \( x \) is the fraction of the first liquid. Table 1 shows refractive indices \( n \) of some laser crystals, glass, and liquids used in this work for the preparation of immersion mixtures. The refractive indices of materials at wavelengths \( \lambda \) of 650 nm and 1060 nm (with 4 decimal places) are given in accordance with the data of website [14]. For several materials, refractive indices are given for \( \lambda = 589 \) nm according to [15–19]. All liquids listed in Table 1 are transparent in the visible and near-IR spectral regions (400–1000 nm).

| Material          | Refractive index |
|-------------------|------------------|
|                   | \( \lambda = 650 \) nm | \( \lambda = 589 \) nm | \( \lambda = 1060 \) nm |
| Acetone           | 1.3574           | 1.3526               |
| Ethanol           | 1.3600           | 1.3548               |
| Heptane           | 1.3872           | 1.3827               |
| LiF               | 1.3910           | 1.3866               |
| Isobutyl alcohol  | 1.3913           | 1.3862               |
| Ethylene glycol   | 1.4296           | 1.4232               |
| Nd:YLiF           | 1.4524(o), 1.4750(e) | 1.4480(o), 1.4702(e) |  |
| Glycerol          | 1.4701           | 1.4631               |
| Toluene           | 1.4926           | 1.4813               |
| Benzene           | 1.4915           | 1.4802               |
| Vaseline oil      |                  | 1.503                |
| Benzyl alcohol    | 1.536            |                     |
| Anise oil         | 1.560            |                     |
| Nd:glass          |                  | 1.55                 |
| NdP5O14           |                  | 1.60                 |
| Cinnamaldehyde    |                  | 1.62                 |

To prepare a slurry index-matched for the wavelength region near the expected operating wavelength of a given laser material, we used the laser setup shown in figure 2. A diode laser (\( \lambda = 650\)nm), a He-Ne laser (\( \lambda = 633 \) nm), and a Nd:YAG laser (\( \lambda = 1064 \) nm) with beam diameters of 1±2 mm were used as reference light sources. To measure the power transmitted through a cuvette with a slurry, a power meter was placed behind an iris diaphragm near the focus of the lens with a focal length \( f \approx 85 \) mm. Liquids were added to the layer of powder (≈ 1 cm in height) in a cuvette using a syringe (dropwise, with stirring), so that a buffer layer of liquid was formed above the slurry. The optimum composition of the immersion liquid was achieved when the reference beam transmission through the slurry was maximum and the transmitted laser beam spot at a remote screen had the best quality. For LiF, the first liquid component with the higher index may be anise oil, vaseline oil, or toluene, and the second component with the lower index may be ethanol, acetone, or heptane. The vaseline oil/heptane and toluene/ethanol mixtures were used as immersion liquids for LiF:F2 and LiF particles. At the optimal composition, the transmission of the He–Ne laser beam through the slurry of a fixed thickness depended on the size of particles. A decrease in the thickness of the layer of particles in the cuvette, as well as the use of grains of larger sizes (500±1000 µm and larger), leads to a reduction of scattering in the medium. For 500±1000-µm LiF or LiF:F2 grains in a 3-
mm-thick cuvette, the fraction of transmitted laser radiation in the angle of 0.01 rad reaches 70%. For particles of smaller sizes (200–500 μm and < 200 μm), the transmission decreases to 30–60%.

Figure 2. Scheme of the laser setup for preparation and testing of the index-matched slurry: (1) laser source; (2) cuvette with the slurry; (3) components for preparing immersion liquids; (4) lens; (5) diaphragm; (6) screen; (7) power meter.

All these observations correspond to the characteristics of the Christiansen filter (CF)—a well-known device in the form of a cuvette with glass or crystal powder in an immersion liquid, which is used to obtain spectral selection of radiation [20, 21]. CF is characterized by a peak of transmission at the operating wavelength. The transmission spectra of LiF and LiF:F₂ slurries with 500–1000; 200–500, and < 200-μm granules were measured on a Cary-5000 spectrophotometer (figure 3). The spectra of LiF:F₂ slurries in the region 300–900 nm are similar to the spectrum of a LiF:F₂ bulk plate (figure 3a). These spectra exhibit the absorption band of F₂ centers near 450 nm and the absorption peak of the immersion liquid near 1200 nm.

Using the data on absorption for the slurry and the pure immersion liquid, it was possible to estimate the particle filling factor in the slurry to be ≈ 0.5. Figure 3(b) shows the spectrum of the slurry of pure LiF in a toluene/ethanol mixture with the transmission maximum near λ = 570 nm. This maximum can be shifted along the wavelength scale by changing the ratio of the immersion liquid components.

Figure 3. Transmission spectra of (a) (1) 2.5-mm-thick LiF:F₂ crystal plate and (2, 3) 3-mm-thick cuvettes with the LiF:F₂ slurry with (2) 500–1000 and (3) 200–500 μm granules in heptane/vaseline oil and (b) the 3-mm-thick cuvette with pure LiF granules (size < 200 μm) in toluene/ethanol mixture.
The calculations using data from [14] showed that the position of this maximum corresponds to the crossing point of the dispersion curves of LiF crystal and toluene/ethanol mixture, which testified that the spectrum in figure 3b is a CF spectrum. At the same time, the spectra of the LiF:F\textsubscript{2} slurry with 200–500 and 500–1000 μm granules do not have this characteristic peak. Perhaps, this feature was smoothed out due to the presence of the strong and wide LiF:F\textsubscript{2} absorption band (figure 3a).

In addition to the LiF:F\textsubscript{2} slurry, we prepared active media based on particles of crystals and glasses doped with Nd\textsuperscript{3+} ions in immersion liquids and studied their characteristics. The preparation of an index-matched slurry of particles of such a well-known laser crystal as Nd:YAG is not an easy task due to the high refractive index (n ≈ 1.82) of this crystal. Particles of another widely used crystal, Nd:YLF, (n ≈ 1.45) were obtained from a bulk Nd:YLF sample. The moderate value of the refractive index of the Nd:YLF crystal allowed us to choose several options for the immersion liquid. However, the photos in figure 4 illustrate the difficulties involved in trying to get an index-matched slurry for Nd:YLF. Figure 4 shows propagation of a He-Ne laser beam through the 3-mm-thick cuvette with a powder of isotropic (cubic) LiF crystal in a mixture of heptane/vaseline oil or through a powder from anisotropic Nd:YLF crystal in an immersion mixture of isobutyl and benzyl alcohols. Sizes of grains were 500–1000 μm in both cases. Comparison of the beam profiles on a remote screen indicates high scattering losses for the cuvette with Nd:YLF powder. The reason of these losses is that the refractive index of the liquid cannot be equal simultaneously to the ordinary and extraordinary indices of anisotropic Nd:YLF particles. Thus, high scattering losses (which may suppress lasing) are expected for slurry with particles of anisotropic materials.

Interesting materials for slurry lasers are highly concentrated Nd\textsuperscript{3+}-doped phosphate glass [15] and neodymium crystals, such as Nd\textsubscript{5}P\textsubscript{5}O\textsubscript{14} [18, 22, 23]. The concentration of Nd\textsuperscript{3+} ions in these media can exceed 10\textsuperscript{21} cm\textsuperscript{-3}, which results in the record laser gains (up to hundreds of cm\textsuperscript{-1} in Nd\textsubscript{5}P\textsubscript{5}O\textsubscript{14} [22]). The refractive indices of these materials are n = 1.5±1.6, which allows using a number of liquids as immersion mixture components. Samples of Nd\textsubscript{5}P\textsubscript{5}O\textsubscript{14} crystal (n ≈ 1.60) and LGSI-50 phosphate Nd:glass (n ≈ 1.55; Nd\textsuperscript{3+} concentration ≈ 12%) were provided by IRE of the RAS. A powder with particle dimensions from 200 to 500 μm was obtained by crushing LGSI-50 glass bulk samples. For experiments with Nd\textsubscript{5}P\textsubscript{5}O\textsubscript{14} crystals, we used 0.2–0.4-mm thick plates with facets up to 2×4 × 1.5 mm taken directly from a growth crucible [23]. Such small crystals can, generally speaking, be considered as components of an active medium for a flow-through slurry-like laser. Because of this, we did not crush these small pieces of the Nd\textsubscript{5}P\textsubscript{5}O\textsubscript{14} crystal into powder.

![Figure 4](image_url)

**Figure 4.** Images of a He-Ne laser beam on a screen at a distance of 1 m from the source in the cases (a) without a cuvette; (b) with a 3-mm thick cuvette with an index-matched slurry of LiF particles, and (c) with a 3-mm thick cuvette with a slurry of anisotropic Nd:YLF particles.
Figure 5 shows the spectra of LGSI-50 Nd:glass powder (a) and a 0.4-mm-thick NdP₅O₁₄ plate (b) placed in a 1-mm-thick cuvette with immersion mixtures of cinnamaldehyde and benzyl alcohol. The immersion compositions for the spectral region near 1060 nm were prepared at a laser stand (figure 2) separately for each material. Both spectra show characteristic absorption bands of Nd³⁺ ions. The transmittance of the cuvettes near 1060 nm reached 80%. The transmission curve of LGSI-50 glass clearly shows an increase in scattering losses in the short-wavelength region (figure 5a). The absorption bands at 1200 and 1500 nm in the spectra correspond to the absorption by the immersion liquid.

![Figure 5](image)

**Figure 5.** Transmission spectra of the cuvette (1-mm gap) with powder of LGSI-50 glass particles sized 200-500 μm (a); and with a 0.4-mm thick NdP₅O₁₄ crystal plate (b) in benzyl alcohol/cinnamaldehyde immersion mixtures.

3. Experiments on excitation of laser oscillation in active media

The experiments on lasing in samples were performed using a pulsed Nd:YAG laser LOTIS TII with conversion of 1060 nm radiation into the 2-nd and 3-rd harmonics. The scheme of the experiment on the LiF:F₂ slurry is shown in figure 6. A transversely pumped Coumarin 120 (C120) dye laser was used as a pump source. The cuvette with C120 dye solution in a resonator of length L ≈ 8 mm was pumped by 12-ns pulses of the 3-rd harmonic (354 nm) of a pulsed LOTIS TII Nd:YAG laser. The energy in a single pump pulse was up to 8.7 mJ. This radiation was focused through the side wall of the cuvette into the dye solution. The C120 dye laser emitted 11-ns pulses with an energy up to 1.6 mJ at wavelength λₚ ≈ 446 nm, which corresponds to the absorption band of F₂ centers (figure 3a). The pump beam diameter was 2 mm. The samples (LiF:F₂ bulk plates or cuvettes with a 2-3-mm gap filled by LiF:F₂ slurry) were placed into a resonator (L ≈ 40 mm) formed by two flat mirrors. The dye laser radiation was focused onto the samples by a lens with f = 172 mm along the resonator axis through the input mirror with a 90% transmission at λₚ and a high reflection (HR = 99%) in the red spectral region. The diameter of the pump spot at the samples was 0.5 mm, and the maximum pump intensity reached 60 MW·cm⁻². The pump radiation was almost completely (> 98%) absorbed in the LiF:F₂ media. Mirrors with transmission from T = 1% to T = 23% at a wavelength near 680 nm were used as an output coupler (OC). 10-ns pulses of red collimated radiation in the wavelength region near 680 nm were produced at the output of the resonator containing LiF:F₂ plates or cuvettes with LiF:F₂ granules.

The pulse energy from all types of LiF:F₂ active elements was measured using a calibrated silicon photodiode FD24 connected to a digital oscilloscope. Figure 7 compares the dependences of the output energy of the LiF:F₂ slurry lasers (for two compositions) and the LiF:F₂ single crystal laser on the pump energy. All measurements were carried out in the same resonator configuration with the OC transmission of 13%.
Figure 6. Scheme of the experiment on pumping the LiF:F\textsubscript{2} slurry active medium by a Coumarin 120 dye laser: (1) Coumarin 120 dye laser, (2) beam of the 3rd harmonic of a pulsed Nd:YAG laser; (3) focusing lens; (4) cuvette with the LiF:F\textsubscript{2} slurry.

From the graphs in figure 7, one can see that the efficiency of the slurry laser is only twice as low as that of the single crystal laser. The maximum output energy of the slurry laser was about 83 µJ at a pump energy of 1.34 mJ; the slope efficiency was 12.9%. The LiF:F\textsubscript{2} slurry laser beam divergence near the generation threshold was estimated to be 2 mrad and increased up to 5 mrad at higher pump energies. It was found that the lasing threshold is smaller and the laser output energy is higher for 500±1000-µm granules as compared with 200±500-µm granules. The output energy of the slurry laser with a 2-mm-thick cuvette was lower than in the case of a 3-mm-thick cuvettes. For particles < 200 µm in size, lasing was not achieved even for the OC with a reflectivity of 99%.

Experiments on excitation of media doped with Nd\textsuperscript{3+} ions were carried out both with powders ( slurries) and with samples of the bulk material in an immersion liquid and without it. NdP\textsubscript{5}O\textsubscript{14} samples with dimensions of (2±4) × (1±1.5) × (0.2±0.4) mm were used for lasing experiments. Despite the NdP\textsubscript{5}O\textsubscript{14} crystal anisotropy, a high gain in this crystal allowed us to expect lasing in these small samples even in conditions of incomplete immersion. The scheme of the experiment on the excitation of lasing in NdP\textsubscript{5}O\textsubscript{14} crystals is shown in figure 8. The active medium was pumped by pulses (20 ns, 5 mJ) of a pyromethene 597 (PM597) dye laser at wavelength λ\textsubscript{p}=581 nm, which coincides with the absorption peak of neodymium in NdP\textsubscript{5}O\textsubscript{14} crystals (figure 5b).

Figure 7. Dependences of the output energy on the pump energy for a laser based on a 2.4 mm thick LiF:F\textsubscript{2} bulk plate and on LiF:F\textsubscript{2} slurry in a 3 mm thick cuvette; for a laser based on 0.4-mm-thick NdP\textsubscript{5}O\textsubscript{14} crystals in a 1-mm-thick cuvette with an immersion liquid; and for a laser based on a 2-mm-thick LGSI-50 glass plate.
Figure 8. Scheme of the experiment on pumping of NdP$_3$O$_{14}$ crystals in a 1-mm-thick cuvette with immersion liquid by a PM597 dye laser.

The dye laser radiation was focused on the cuvette with 1- or 2-mm gap by a 13-cm focus lens. A semiconfocal resonator with $L = 2.5$ cm was used: a HR spherical mirror with a radius of 5 cm and a flat output mirror with 1.7% transmission at the lasing wavelength, which is 1051 nm for NdP$_3$O$_{14}$ crystal. The cuvette was located close to the flat mirror. The residual pump radiation passed through the cuvette and the output mirror was cut off from the generation by two IR filters. Figure 7 shows the dependences of the output energy on the pump energy for the lasers based on one and two 0.4-mm-thick NdP$_3$O$_{14}$ samples in a cuvette with benzyl alcohol/cinnamaldehyde immersion; oscillograms of the pump and NdP$_3$O$_{14}$ laser pulses are shown in figure 9.

In order to verify the lifetime of Nd$^{3+}$ ions at the upper $^4F_{3/2}$ laser level in LGSI-50 glass, an experiment was carried out to measure this parameter. Luminescence in a polished LGSI-50 glass plate was excited by pulses of the 2-nd harmonic of a Nd:YAG laser. Radiation at the 1.06 μm $^4F_{3/2} \rightarrow ^4I_{11/2}$ transition was recorded using a VM-25 monochromator. An FEU-62 photomultiplier tube and a Tektronix TDS-2022 digital storage oscilloscope recorded the luminescence dynamics. The obtained dependence is well described by an exponential function. The lifetime of the upper laser level for the LGSI-50 glass was found to be 45 μs. The experiments on lasing with LGSI-50 glass samples were carried out according to a scheme similar to the experimental setup for NdP$_3$O$_{14}$ crystals (figure 8). The dependence of the output energy of a polished 2-mm-thick LGSI-50 glass plate (without immersion) on the pump energy is shown in figure 7. The energy of 10-ns pulses of the LGSI-50 glass laser reached 150 μJ. Pieces of LGSI-50 glass (0.8–2.0 mm thick) with different classes of surface finish were placed in cuvettes with a 2–3-mm gap filled with an cinnamaldehyde/benzyl alcohol immersion mixture. Laser pulses with an energy of up to 100 μJ were obtained in the same resonator configuration for all these samples in immersion, even for pieces with matted surfaces.

Figure 9. Oscillograms of the dye laser pulse and the pulse of the NdP$_3$O$_{14}$ laser.

However, when cuvettes with LGSI-50 glass microparticles in immersion were installed in the
resonator, the lasing threshold was not reached due to scattering losses. Similar results were obtained with particles of highly concentrated Nd-doped phosphate glass [15]. The source of losses in the glass powder slurry in addition to incomplete immersion can be various centers of scattering on the surface and inside the particles. Note that comparison of the transmittance of a 1±3-mm-thick cuvettes with an immersion mixture and crushed glass particles (T\(_{1.06} = 60\pm80\%\)) with the transmittance of the same cuvettes with polished glass beads (without activator) 0.7±0.8 mm in diameter showed the advantage of the glass beads (T\(_{1.06} = 90\%\)). This provides grounds for fabricating Nd glass beads and conducting lasing experiments with them.

4. Conclusion

Active media based on particles of solid-state laser materials distributed in a liquid carrier may bring new solutions to the problem of heat removal in lasers due to active media circulation. The lasing characteristics of several active media based on particles of laser glass and crystals in an immersion liquid were measured upon pumping by nanosecond laser pulses in the gain switching regime. The technique of preparation and characterization of dense index-matched mixtures of solid-state microparticles and an immersion liquid (slurry) was tested for particles of LiF crystals with F\(_2\) CCs, as well as for particles of laser glass and crystals doped with Nd\(^{3+}\) ions. The observed nanosecond pulses of LiF:F\(_2\) emission near 680 nm in collimated beams from cuvettes with the slurry in a resonator and the dependences of the output energy on the pump energy give grounds for the conclusion that lasing from a dense active medium based on microparticles of a solid-state laser material in an immersion liquid (a slurry laser) was demonstrated. For cuvettes with Nd:glass powders in an immersion liquid installed in a resonator, the lasing threshold was not reached due to high scattering losses and insufficient gain. Lasing was obtained on unpolished samples of concentrated neodymium phosphate glass and on small neodymium pentaphosphate crystals in immersion liquids. The continuation of the study on the generation and amplification of cw and pulsed laser radiation in slurry-like active media seems reasonable. It is of interest to conduct experiments with such active media based on standardized particles, such as glass beads highly doped with Nd or Yb ions. Experiments with particles of LiF crystals with color centers should also be continued.

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