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Laser materials search and characterization

Maxim E. Doroshenko

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<ul> <li>14. ABSTRACT Thus, all the tasks of the project have been fulfilled. Task 1. Single crystals of doped and pure halides are synthesized and their optical properties are studied. Preforms are made and crystalline waveguides are produced on their basis. Task 2. Crystalline waveguides of the composition AgBr/AgBr doped with dysprosium ions are obtained by extrusion, and their optical and spectral properties are studied. Task 3. Waveguides based on LiF fluoride are obtained by extrusion for the first time. Task 4. Spectroscopic properties of Dy3+ ions in chalcogenide crystals and fibers are studied. Task 5. Crystals and ceramics doped with rare-earth ions, as well as glasses and crystals doped with bismuth ions, are synthesized. Their spectroscopic and lasing properties are studied. Task 6. New technologies are developed and various waveguiding structures (waveguides and planar waveguides) based on chalcogenide and fluoride crystalline and ceramic materials are obtained. The optical and lasing properties are studied. 15. SUBJECT TERMS</li></ul>							
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# **ISTC Project No. 4076**

# Laser materials search and characterization

# **Final Project Technical Report**

#### on the work performed from October 01, 2010 to March 31, 2014

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# **1.** Brief description of the work plan: objective, expected results, technical approach

The aim of the project was to search and characterize new laser materials in the form of bulk single crystals, ceramics, and fibers. The project includes the study of both single crystals and polycrystalline materials, as well as of various waveguiding structures based on these materials, including ceramic and glass fibers made by extrusion. These materials are of interest in both pure form and doped with various rare-earth ions emitting in the range 1--6  $\mu$ m. The optical, spectral and fluorescence characteristics of the obtained materials are studied in the course of the project. The optical quality of the materials under study was expected to allow one to measure the optical gain and laser properties upon pumping by different sources. The project also includes the study of the spectral, fluorescent, and laser properties of new promising materials (glasses, crystals, and ceramics) doped with bismuth as a new fluorescent ion.

As a result of the project, it was expected to obtain new materials with better properties than that of available analogues and achieve radiation in new spectral regions.

Within the project, it was proposed to perform a search for new crystals, ceramics, and fibers doped with various ions emitting in different regions, to develop the technology of their synthesis, to synthesize these materials, and to study their properties. Stationary and time-resolved spectroscopy was suggested to be used for studying the spectroscopic properties of rare-earth ions in these materials.

Based on the synthesized and studied materials, it was proposed to study the possibility of fabricating structures with waveguiding properties by, in particular, extrusion from singlecrystalline preforms, and to investigate the properties of such waveguiding structures.

#### 2. Method, Experiments, Theory etc.

To investigate the spectroscopic properties of different rare-earth and Bi ions, various methods were used including concentration series, measurements of absorption spectra at different temperatures, stationary and non-stationary fluorescence spectroscopy, time resolved spectroscopy, fluorescence and decay measurements in the broad temperature range 77-300 K. For synthesis and annealing of fluoride single crystals doped with rare-earth ions, a special complex for single crystals growth using Bridgeman technique was applied. The ZnWO<sub>4</sub> crystals were synthesized in platinum crucibles using the modified Stepanov technique in air. Halide crystals were grown in modified installation using melting zone technique. For fluoride fibers extrusion special chamber of titanium alloys and a drawing nozzle of tungsten carbide were specially developed. For fluoride ceramics hot formation from single crystals, a special pressing equipment was used. A special complex was applied for X-ray structural analysis. For spectroscopic measurements with selective excitation, a unique laser spectroscopic complex MALSAN based on solid-state lasers with color centers operating at room temperature was used. This complex allows us to obtain selective excitation of electronic levels of doping ions by nanosecond pulses tunable within the 0.8-1.3 µm spectral range. To register stationary and non-stationary fluorescence, different equipment including various detectors, multichannel photon counters, digital oscilloscopes Tektronix with a 300-MHz bandwidth, boxcar integrators, synchronous amplifiers was applied. The signals were registered in the digital form using a PC. In addition to unique color-centers tunable lasers, the team used other laser excitation sources: CW and pulsed neodymium lasers with frequency conversion to the second, third, and fourth harmonics; tunable CW and pulsed dye lasers; tunable titanium sapphire and alexandrite lasers; diode lasers with output powers up to 30 W with oscillation wavelengths of

795, 805, 960 nm. For low temperature measurements within the 12-300K range, cryogenic equipment from "Oxford Instruments" and "Leybold Gmbh" is available.

#### **3. Results**

In the course of the project, 50AgCl-50AgBr single crystals have been synthesized, both pure and doped with optically inactive (lead) and active (dysprosium) ions. The Judd-Ofelt analysis of the AgBr<sub>0.5</sub>Cl<sub>0.5</sub>:Dy crystal absorption spectrum is performed. The spontaneous lifetimes of most important multiplets are determined. The luminescence spectra of the obtained crystals are measured upon excitation by a neodymium YAG laser at a wavelength of 1.319  $\mu$ m. The luminescence kinetics of dysprosium ions in the obtained crystals are studied in detail. It is found that cross-relaxation occurs between some multiplets. Analysis showed that this is explained by clusterization of Dy<sup>3+</sup> ions. To clarify the nature of the active Dy<sup>3+</sup> centers and reduce possible clusterization, a series of Dy<sup>3+</sup>:AgCl-AgBr crystals co-doped with bivalent Pb<sup>2+</sup> and Bi<sup>2+</sup> ions was synthesized and their luminescent properties were studied. The synthesized crystals were used as performs for extrusion of ceramic fibers. New technological equipment for this process was developed, and the technological parameters were optimized. As a result, pure and doped 50AgCl-50AgBr fibers were obtained.

High-quality LiF crystals were synthesized and a set of experiments was performed on extrusion of fluoride crystals as a base for doped crystalline laser fibers. For this purpose, a special chamber of titanium alloys and a drawing nozzle of tungsten carbide were made. We managed to perform extrusion of fluoride crystals with a low melting temperature for the first time and obtain a 12-fold decrease in the diameter per one process. In addition, combined extrusion of solid solutions of silver halides with polycrystalline lithium fluoride was studied.

Bismuth-doped glasses of the composition BaO--B<sub>2</sub>O<sub>3</sub> are synthesized and their absorption spectra and luminescence spectra excited by radiation with different wavelengths are measured. To study the effect of the synthesis conditions on the formation of different optical centers of bismuth, the samples were synthesized in different atmospheres (oxidizing, neutral, and reducing) It was shown that the samples obtained in the reducing atmosphere exhibit the most intense fluorescence in the near IR region. The luminescence decay kinetics was studied in detail and the lifetimes of the excited levels for different types of bismuth centers were measured. It is shown that the studied glasses contain at least three types of optical centers of bismuth ions luminescent in the visible spectral region and two types luminescent in the near-IR region. The dependence of the luminescent properties of bismuth in borate glasses on the content of aluminum oxide was studied. Several samples of Bi-doped borate glasses were specially grown in a graphite crucible with addition of different Al<sub>2</sub>O<sub>3</sub> concentrations. It was found that an increase in the Al<sub>2</sub>O<sub>3</sub> concentration in the glass composition enhances the formation of Bi centers with the short-wavelength IR fluorescence at 1150 nm. The fluorescence spectra of these samples were also compared with the spectra of Bi-doped B<sub>2</sub>O<sub>3</sub>--BaO glasses synthesized in corundum (Al<sub>2</sub>O<sub>3</sub>) crucibles, which allowed us to determine the uncontrolled enrichment of these glasses with aluminum oxide occurring upon melting.

BaF<sub>2</sub> single crystals were grown by a classical technology with doping with BiF<sub>3</sub> in a fluorinating atmosphere. It was found that these crystals exhibit only fluorescence of trivalent bismuth in the region of 400--500 nm, while fluorescence in the IR region is absent. The new samples of Bi:BaF<sub>2</sub> crystals were grown using Bi<sub>2</sub>O<sub>3</sub> as a raw material instead of BiF<sub>3</sub>. These us-grown crystals demonstrated luminescence in the red spectral region and a weak IR luminescence of bismuth ions in region of 1.1  $\mu$ m. The combined effect of annealing and electron-beam irradiation of bismuth-doped barium fluoride crystals on the IR luminescence parameters was studied. A significant increase in the luminescence intensity was obtained in the samples irradiated by a 150-keV electron beam and preliminarily annealed at a temperature of 1200°C. Detailed investigation of the luminescence decay kinetics and time-resolved

luminescence spectra allowed us to separate four different optical centers of bismuth ions in barium fluoride crystals with different lifetimes and determine their contributions to the total spectrum. Irradiation of bismuth-doped barium fluoride crystals with a much higher energy (20 MeV) led to an even higher luminescence intensity at a wavelength 1100 nm mainly due to a considerable (by an order of magnitude) increase in the contribution made by the bismuth optical centers with a large (2.5 ms) lifetime. It was also shown that this irradiation leads to the formation of new bismuth centers emitting in the important IR region near 1600 nm. To deeper understanding of the nature of bismuth centers, the fluorescence of Bi<sub>2</sub>O<sub>3</sub>:BaF<sub>2</sub> crystals was studied at the liquid nitrogen temperature (77 K). The decomposition of the spectra into Gaussian components, the time-resolved fluorescence measurements, and the study of the fluorescence decay at low temperatures showed that the long-wavelength fluorescence centers of Bi ions have a different nature than the optical centers responsible for the red and near IR fluorescence.

The optical centers of Bi ions were also studied in CsI crystals, which were specially grown within the framework of the project. It was shown that the formation of Bi optical centers in CsI crystal strongly depends on the synthesis conditions. It was found that the fluorescence intensity at the desired wavelength of 1600 nm considerably increases after irradiation by a high-energy electron beam. The performed measurements of the fluorescence spectra and fluorescence decay at room and liquid nitrogen temperatures showed that the Bi optical centers in BaF<sub>2</sub>:Bi<sub>2</sub>O<sub>3</sub> and CsI crystals have much in common.

Bismuth ions were also studied in zinc tungstate single crystals doped with different concentrations of bismuth ions, which were specially grown by the Czochralski method. Detailed experimental studies of their absorption, luminescence, and luminescence excitation spectra, as well as the investigations of the luminescence decay kinetics at room and liquid nitrogen temperatures and comparison of the obtained results with the data determined previously for other crystals, allowed us to establish for the first time that the bismuth ions in zinc tungstate crystals form only two optical centers, which have lifetimes of 1 and 6  $\mu$ s at room temperature and are responsible for the luminescence band in the IR spectral region. It is shown that, at liquid nitrogen temperature, the luminescence of the short-lived center is dominant. The existence of only two luminescent centers of bismuth ions at room temperature and of one center at 77K makes this crystal most convenient for investigations of the optical properties of bismuth ions

Single crystals of CaF<sub>2</sub>, SrF<sub>2</sub>, BaF<sub>2</sub> and their solid solutions doped with rare-earth neodymium, thulium, holmium, and erbium ions are synthesized by the Bridgman method. Doped fluoride optical nanoceramics of similar compositions is obtained from these single crystals by hot forming technique, which was developed and optimized within the framework of the project. The spectroscopic and lasing properties of the grown CaF<sub>2</sub> crystals and CaF<sub>2</sub>-SrF<sub>2</sub> and BaF<sub>2</sub>-SrF<sub>2</sub> solid solutions doped with thulium or holmium ions, as well as co-doped with Tm<sup>3+</sup> and Ho<sup>3+</sup> ions, were studied in detail. It is shown that the maxima of the thulium and holmium absorption and luminescence spectra shift to longer long wavelengths in the sequence Ca-Sr-Ba. Upon excitation of Tm<sup>3+</sup> by a laser diode with a wavelength of 790 nm, tunable lasing of Tm<sup>3+</sup> was obtained in a nonselective cavity in the region of 1.9  $\mu$ m with the maximum slope efficiency up to 23% and lasing of holmium ions was obtained in the region of 2.1  $\mu$ m with a slope efficiency up to 17%.

New samples of calcium fluoride ceramics doped with erbium ions with a good optical quality were produced and their luminescent properties were studied. Detailed study of the luminescence decay kinetics showed the occurrence of efficient cross relaxation from the lower laser level, which makes it possible to obtain intense luminescence both in the near IR and in the visible wavelength regions. Upon pumping by a laser diode at a wavelength of 974 nm, lasing was achieved in nonselective and selective cavities with slope efficiencies of 2.2% and

1.5%, respectively. In a cavity with a Lyot filter, tunable lasing was achieved within the range 2600--2755 nm.

A new technology of hot formation was developed to produce composite structures possessing the properties of weakly guiding waveguides based on fluoride materials with an undoped cladding and a doped core. Using this technology, we made samples of planar waveguides with a core of a  $SrF_2:NdF_3$  ceramic plate and two cladding plates made of  $SrF_2$  single crystals, as well as samples of a crystalline planar waveguide structure of the composition  $CaF_2-Nd^{3+}:CaF_2-CaF_2$ . The waveguiding properties of these composite structures were demonstrated and their lasing properties were studied under diode pumping. The lasing wavelength in the studied planar waveguides is found to be close to the wavelength of corresponding bulk materials.

#### 4. Conclusion

Thus, all the tasks of the project have been fulfilled.

Task 1. Single crystals of doped and pure halides are synthesized and their optical properties are studied. Preforms are made and crystalline waveguides are produced on their basis.

Task 2. Crystalline waveguides of the composition AgBr/AgBr doped with dysprosium ions are obtained by extrusion, and their optical and spectral properties are studied.

Task 3. Waveguides based on LiF fluoride are obtained by extrusion for the first time.

Task 4. Spectroscopic properties of  $Dy^{3+}$  ions in chalcogenide crystals and fibers are studied. Task 5. Crystals and ceramics doped with rare-earth ions, as well as glasses and crystals doped with bismuth ions, are synthesized. Their spectroscopic and lasing properties are studied.

Task 6. New technologies are developed and various waveguiding structures (waveguides and planar waveguides) based on chalcogenide and fluoride crystalline and ceramic materials are obtained. The optical and lasing properties are studied.

#### Attachment 1:

#### List of published papers and reports with abstracts

1. Jan Sulc, Michal Nemec, Richard Svejkar, Helena Jelinkova, Maxim E. Doroshenko, Pavel P. Fedorov, and Vyacheslav V. Osiko, Diode-pumped  $Er:CaF_2$  ceramic 2.7 µm tunable laser, Optics Letters, Vol. 38 (17), pp. 3406-3409 (2013).

#### Abstract

Spectroscopic and laser properties of a newly developed high optical quality  $Er:CaF_2$  hotformed ceramic were investigated. Under pulsed 968 nm laser diode pumping, the mid-infrared (2.7 µm) radiation was obtained with a slope efficiency of 3%. Laser tunability was reached using a birefringent filter and the laser tuning range of 118 nm, from 2687 up to 2805 nm, was demonstrated. The maximal output energy reached was 0.48 mJ at 2730 nm for the absorbed pumping energy 34 mJ.

http://www.opticsinfobase.org/ol/abstract.cfm?uri=ol-38-17-3406

2. M.Sh. Akchurin, T.T. Basiev, A.A. Demidenko, M.E. Doroshenko, P.P. Fedorov, E.A. Garibin, P.E. Gusev, S.V. Kuznetsov, M.A. Krutov, I.A. Mironov, V.V. Osiko, P.A. Popov, CaF<sub>2</sub>:Yb laser ceramics, Optical Materials, Vol. 35 (3), pp. 444-450 (2013). Abstract

CaF<sub>2</sub>:Yb fluoride laser ceramics, prepared by hot-forming, exhibit the same optical properties as starting single crystals. Slope efficiency of the Ca<sub>0.95</sub>Yb<sub>0.05</sub>F<sub>2.05</sub> is equal to 35% in the pulsed mode of laser operation. Decrease of ytterbium concentration in CaF<sub>2</sub>:Yb samples down to 3 mol.% resulted in the essential improvement of Ca<sub>0.97</sub>Yb<sub>0.03</sub>F<sub>2.03</sub> thermal conductivity from 3.5 to 4.5 W/m K, but slightly decreased (down to 30%) slope efficiency of the samples under both pulsed and CW mode of operation. Alternative hot-pressing synthesis of CaF<sub>2</sub>:Yb fluoride laser ceramics provided materials with superior mechanical properties (microhardness H = 3.2 GPa and fracture toughness  $K_{1C} = 0.65$  MPa m<sup>1/2</sup>) in comparison with hot-formed and/or single crystal CaF<sub>2</sub>:Yb specimens. For the first time, lasing has been observed for the novel aforementioned hot-pressed CaF<sub>2</sub>:Yb ceramics.

http://www.sciencedirect.com/science/article/pii/S0925346712004405

3. Doroshenko M.E., Demidenko A.A., Fedorov P.P., Garibin E.A., Gusev P.E., Jelinkova H., Konyshkin V.A., Krutov M.A., Kuznetsov S.V., Osiko V.V., Popov P.A., Shulc J. Progress in fluoride laser ceramics. // Phys. Stat. Solidi C, 10(6), 952-957(2013). Abstract

A variety of  $Er^{3+}$ ,  $Tm^{3+}$ , and  $Yb^{3+}$ -activated  $CaF_2$ -,  $SrF_2$ -,  $(Ca, Sr)F_2$ - and  $(Sr, Ba)F_2$ -based fluoride laser ceramics have been prepared by the hot-forming technique. Lasing of the  $SrF_2$ :  $Yb^{3+}$ ,  $(Ca, Sr)F_2$ :  $Er^{3+}$ ,  $(Ca, Sr)F_2$ :  $Tm^{3+}$ ,  $(Ba, Sr)F_2$ :  $Tm^{3+}$  and  $(Ba, Sr)F_2$ :  $Yb^{3+}$  specimens with diode pumping has been observed for the first time. Thermal conductivity data for the above fluorite-type laser materials are also presented and discussed.

http://onlinelibrary.wiley.com/doi/10.1002/pssc.201300023/abstract

4. Basiev, T. T.; Basieva, I. T.; Doroshenko, M. E., Luminescent nanophotonics and advanced solid state lasers, JOURNAL OF LUMINESCENCE, Vol. 133, pp. 233-243, 2013. Abstract

In this review, authors present their latest findings in luminescence quenching kinetics theory and advanced solid state laser experiments. Luminescence quenching kinetics is a popular and exceptionally useful tool to analyze the nanosized luminophores and laser material nanostructure. Quenching kinetics may be multistage, some stages having a complex, not exponential, form. It is often the case for modern laser materials, which are nanostructurized, and for particular cases of energy transfer (such as cooperative down-conversion). We present compact and easy-to-use analytical expressions and computer simulation for various cases of nonexponential quenching kinetics: migration-accelerated quenching in bulk material; cooperative luminescence quenching in bulk material; and two extreme cases of energy transfer in nanoparticles – static and with superfast migration (both including cooperative case of luminescence quenching in ensembles of acceptors comprised of two-, three-, and more particles). We also review the most perspective laser experiments lately performed in our laboratory, including those on fluoride laser nanoceramics and materials for middle infra-red lasers.

http://www.sciencedirect.com/science/article/pii/S0022231311007137.

5. Konyushkin, V. A.; Nakladov, A. N.; Konyushkin, D. V.; Doroshenko M.E. et al., Ceramic planar waveguide structures for amplifiers and lasers, QUANTUM ELECTRONICS, Vol. 43(1), pp. 60-62, 2013..

#### Abstract

Ceramic and crystalline weakly guiding optical fibres with the core – cladding refractive index difference of  $10^{-2} - 10^{-4}$  are fabricated by a hot pressing method. The waveguides with one or several cores for operation in the spectral range  $0.2 - 5 \mu m$  are produced. The waveguides are based on CaF<sub>2</sub>, SrF<sub>2</sub>, and BaF<sub>2</sub> ceramics and crystals and their solid solutions doped with trivalent Pr, Nd, Tb, Dy, Yb, Ho, Er, and Tm ions, as well as on LiF ceramics and crystals with colour centres. The first results of investigation of the lasing properties of ceramic SrF<sub>2</sub>: NdF waveguides under diode pumping are presented, and the prospects of further investigation are discussed.

http://www.quantum-

electron.ru/php/paper\_rus.phtml?journal\_id=qe&paper\_id=15006&year\_id=2013&volume=43 &issue\_id=1&fpage=60&lpage=62

#### Attachment 2:

#### List of presentations at conferences and meetings with abstracts

1. Konstantin K. Pukhov, Tasoltan T. Basiev, Chang-Kui Duan. Luminescent properties of doped dielectric nanocrystals, 15th International Conference "Laser Optics 2012". St.Petersburg, June 25-29, 2012. Technical Program, p. 50, paper ThR6-47. Abstract

# Here we present the results of our theoretical study of the optical characteristics (lifetimes, IR absorption coefficient, Raman gain coefficient) for small-radius optical centers in subwavelength spherical and ellipsoidal nanocrystals embedded in a dielectric medium.

2. M.E. Doroshenko, T.T. Basiev, V.A. Konyushkin, V.V. Osiko, H. Jelinkova, J. Sulc, Laser properties of  $\text{Er}^{3+}$  ions in CaF<sub>2</sub> and CaF<sub>2</sub>-SrF<sub>2</sub> fluoride ceramics, 15<sup>th</sup> International Conference Laser Optics 2012, St.Petersburg, Russia, June 25 - 29, 2012, paper FRR1-42. Abstract

Lasing of  $Er^{3+}$  ions in CaF<sub>2</sub> and CaF<sub>2</sub>-SrF<sub>2</sub> ceramics under laser diode pumping was realized with slope efficiency up to 2.2%. Using Lio filter tuning within 2700-2760 nm spectral range was obtained

3. K.K. Pukhov. Radiative characteristics of the doped nanocrystals, 3rd International Conference on the Physics of Optical Materials and Devices (ICOM 2012). Belgrade, Serbia, 3-6 September 2012. Book of Abstracts, p. 259.

#### Abstract

We present results of the theoretical study of optical characteristics for the small-radius optical centers in the subwavelength ellipsoidal nanocrystals embedded in a medium. Analytical expressions are obtained for the electric-dipole and magnetic-dipole decay rates of excitations of optical centers in a magnetodielectric nanocomposite.

4. J.Sulc, M.E.Doroshenko, H.Jelinkova, T.T.Basiev, V.A.Konyushkin, V.V.Osiko, Tunability of laser based on Yb-doped hot-pressed CaF<sub>2</sub> ceramics, Laser Sources and Applications, Proceedings of SPIE Vol.8433, p. 84331P-8, 2012.

#### Abstract

The aim of presented study was an investigation of tunability of diode pumped laser based on hot-pressed Yb:CaF<sub>2</sub> ceramics. The tested Yb:CaF<sub>2</sub> sample was in the form of 3.5mm thick plane-parallel face-polished plate (without AR coatings). The Yb<sup>3+</sup> concentration was 5.5 %. A fiber (core diameter 200 µm, NA= 0.22) coupled laser diode (LIMO, HLU25F200-980) with emission at wavelength 976 nm, was used for longitudinal Yb:CaF<sub>2</sub> pumping. The laser diode was operating in the pulsed regime (4 ms pulse length, 20 Hz repetition rate). The duty-cycle 8% ensured a low thermal load even under the maximum diode pumping power amplitude 10W (crystal sample was only air-cooled). This radiation was focused into the crystal (pumping beam waist diameter ~ 170 µm). The 145mm long semi-hemispherical laser resonator consisted of a flat pumping mirror (HR @ 1.01 – 1.09 µm, HT @ 0.97 µm) and curved (r = 150mm) output coupler with a reflectivity of ~ 98% (a, 1.01 – 1.09 µm. Tuning of the ytterbium laser was accomplished by using a birefringent filter (single 1.5mm thick quartz plate) placed inside the optical resonator at the Brewster angle between the output coupler and the laser active medium. The extremely broad and smooth tuning was obtained. The laser was continuously tunable over ~ 66nm (from 1015nm to 1081 nm) and the tuning band was mostly limited by free spectral range of used birefringent filter. The tunability FWHM was 40 nm corresponding bandwidth 10 THz results in Fourier limited gaussian pulse width ~ 40 fs (FWHM). The maximum output power amplitude 0.68W was obtained at wavelength 1054nm for absorbed pump power amplitude 6W. The laser slope efficiency was 15%

5. Maxim E. Doroshenko, Vasilii Konyushkin, Alexander Karasik, Pavel Fedorov, Vyacheslav Osiko, Helena Jelínková, Jan Šulc, Fluoride Ceramics for Mid IR Lasing (2-3 μm) and Planar

Waveguides, Advanced Solid-State Lasers Congress 2013, October 17- November 1, Paris, France, paper AMA1.6, 2013.

#### <u>Abstract</u>

Fluoride ceramics doped with  $\text{Er}^{3+}$ ,  $\text{Tm}^{3+}$ ,  $\text{Ho}^{3+}$  ions was developed and its spectroscopic and laser properties in 2-3  $\mu$ m spectral region were investigated. Slope efficiency about 20% was demonstrated in 2  $\mu$ m spectral region.

6. Jan Sulc, Michal Nemec, Helena Jelinkova, Maxim E. Doroshenko, Pavel Fedorov, Vyacheslav Osiko, Resonantly Diode Pumping of  $Tm:CaF_2$  and  $Tm:Ho:CaF_2$  Lasers, Advanced Solid-State Lasers Congress 2013, October 17- November 1, Paris, France, paper AM4A.26, 2013.

#### Abstract

The  $\text{Tm}:\text{CaF}_2$  and  $\text{Tm}:\text{Ho}:\text{CaF}_2$  ceramics were investigated as a gain medium in resonantly diode pumped laser. The low-threshold laser emission was obtained at 2027 nm ( $\text{Tm}:\text{CaF}_2$ ) and 2087 nm ( $\text{Tm}:\text{Ho}:\text{CaF}_2$ ).

7. Nemec M., J. Sulc, R. Svejkar, H. Jelinkova, M.E. Doroshenko, V.A. Konyushkin, V.V. Osiko, Spectroscopics and laser properties of  $Er^{3+}$ -doped fluoride laser ceramics, 22<sup>th</sup> International Laser Physics Workshop, Prague, Czech Republic, July 15 – 19, 2013, paper P1.7.

#### Abstract

The spectroscopic and laser properties of newly developed high-optical-quality  $Er:CaF_2$  hotpressed ceramics were investigated. Under pulsed 968nm laser diode pumping the mid infrared (2.7 µm) radiation was obtained with slope efficiency of 3 %. The laser tunability using birefringent filter was reached. The laser tuning range of over 118nm extended from 2687nm to 2805 nm. The maximal output energy reached was 0.48mJ at 2730nm for the absorbed pumping energy 34 mJ.

8. Osiko V.V., P.P. Fedorov, M.E. Doroshenko, E.A. Garibin, Fluoride laser ceramics, Conference on Lasers, Applications, and Technologies (LAT), June 18–22, 2013, Moscow, Russia, paper LTuA1.

#### Abstract

In this presentation we discuss our resent results in developing fluoride laser ceramics. Some features and problems of preparation of the fluoride ceramics, their mechanical, thermal, optical, spectroscopic, and laser properties will be described

9. Fedorov P., Doroshenko M., Garibin E., Osiko V. PROGRESS IN FLUORIDE LASER CERAMICS. 17-th European Symposium on Fluorine Chemistry. Paris, July 21-25. - 2013. Book of Abstracts. - P. 171.

#### Abstract

The history of fluoride laser ceramics begins in 1964, but it was the preparation of fluoride laser ceramics via the hot forming technique that provided a tremendous boost in this area of research and manufacturing. This method is based on the pressure deformation of the single crystals at higher temperatures. Application of this technique made available lasing ceramic fluoride matrixes  $CaF_2$ ,  $SrF_2$ ,  $(Ca,Sr)F_2$ ,  $(Sr,Ba)F_2$  doped with  $Yb^{3+}$ ,  $Nd^{3+}$ ,  $Pr^{3+}$ ,  $Tm^{3+}$ , and  $Er^{3+}$ , including  $CaF_2$ :Yb ceramics. The latter samples possess spectral (including luminescent) and lasing properties similar to that of single crystals. At the same time, the use of an alternative classic technique of hot pressing of powder precursors resulted in significant improvement of the mechanical properties of prepared laser ceramics, while maintaining the same lasing, spectral and other qualities, such as thermal conductivity, thermal expansion, heat capacity, microstructure, etc.

10. V.A. Konyushkin, A.N. Nakladov, D.V. Konyushkin, M.E. Doroshenko, V.V. Osiko, A.Ya. Karasik, Ceramic planar waveguide structures for amplifiers and lasers, Conference on

Lasers, Applications, and Technologies (LAT), June 18-22, 2013, Moscow, Russia, paper LWG5.

#### Abstract

Based on fluoride crystals and ceramic planar weakly guiding waveguides are fabricated by hot pressing technique. The structure of waveguides is demonstrated. Prospects of investigations are discussed.

11. O.K. Alimov, A.G. Papashvili, M.E. Doroshenko, I.S. Voronina, L.I. Ivleva, V.A. Konyushkin, A.N. Nakladov, V.V.Osiko, Investigation of infrared bismuth fluorescence in  $ZnWO_4$  and  $BaF_2$  crystals, Conference on Lasers, Applications, and Technologies (LAT), June 18–22, 2013, Moscow, Russia, paper LWJ39.

#### Abstract

IR fluorescence of bismuth doped as grown  $ZnWO_4$  crystal was investigated under different excitation wavelengths. IR fluorescence with spectral shape and position close to  $BaF_2$ :Bi crystal was observed and characterized.

12. J. Sulc, M. Nemec, H. Jelinková, M.E. Doroshenko, P.P. Fedorov, V.V. Osiko, Diode pumped tunable lasers based on  $Tm:CaF_2$  and  $Tm:Ho:CaF_2$  ceramics, Photonics West, Solid State Lasers XXIII: Technology and Devices, San Francisco, California, USA, 2–4 February 2014.

#### Abstract

The Tm:CaF<sub>2</sub> (4 % of TmF<sub>3</sub>) and Tm:Ho:CaF<sub>2</sub> (2 % of TmF<sub>3</sub>, 0.3 % of HoF<sub>3</sub>) ceramics, prepared using hot pressing, and hot formation technique had been used as an active medium of diode pumped mid-infrared tunable laser. A fibre (core diameter 400 um, NA=0.22) coupled laser diode (LIMO, HLU30F400-790) was used to longitudinal pumping. The laser diode was operating in the pulsed regime (6 ms pulse length, 10 Hz repetition rate) at wavelength 786 nm. The duty-cycle 6 % ensures a low thermal load even under the maximum pumping power amplitude 25 W (ceramics samples were only air-cooled). The 80 mm long semi-hemispherical laser resonator consisted of a flat pumping mirror (HR @ 1.85-2.15 um, HT @ 0.78 um) and a curved (r=150 mm) output coupler with a reflectivity of 98 % @ 1.85-2.0 um for Tm:CaF<sub>2</sub> laser or 99.5 % @ 2.0-2.15 um for Ho:Tm:CaF<sub>2</sub>. Tuning of the laser was accomplished by using a birefringent filter (single 1.5 mm thick quartz plate) placed inside the optical resonator at the Brewster angle. The obtained Tm:CaF<sub>2</sub> tunability ranged from 1892 to 1992 nm (the maximum output energy 1.8 mJ was reached at 1952 nm for absorbed pumping energy 78 mJ). In case of Tm:Ho:CaF<sub>2</sub> laser tunability from 2016 to 2111 nm was reached (the maximum output energy 1.5 mJ was reached at 2083 nm for absorbed pumping energy 53 mJ). Both these material are good candidates for a future investigation of high energy, ultra-short, laser pulse generation.