Synthesis and investigation of highly transparent ceramics

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Abstract. A short review of research works carried out in the Institute of Electrophysics UrB RAS on the development of synthesis technology for laser ceramics and the analysis of their characteristics is presented. These ceramics are widely used as active media of solid-state lasers, transparent armor, envelopes of high intensity discharge lamps, heat-resistant and mechanically strong windows etc.

1. Preparation of nanopowders

Fabrication technology of laser ceramics can be conveniently divided into three stages: synthesis of nanopowders, their compaction and sintering of compacts. The main requirements to nanopowders are small particle size, low degree of agglomeration, high purity and cubic phase. Nanopowders synthesized in a laser plume formed under the action of radiation onto target are more completely respond these requirements. We have created the laser synthesis technology of high purity nanopowders with an average particle size of ~10 nm, a size distribution of 5–45 nm (Figure 1) and an output rate up to 80 g/h. The main characteristics of a laser plume have been investigated and the reasons for appearance of large ~100-μm-sized shapeless particles and spherical particles with a diameter of ~1 mm have been determined as well as the methods of nanopowders separation and collecting have been developed [1].

![Figure 1. Size distribution and TEM image of nanoparticles synthesized in a laser plume](image-url)
2. Synthesis and characteristics of highly transparent ceramics

It has been shown that the relative density of compacts is practically independent of compaction method (uniaxial static pressing with or without ultrasonic action, pulsed magnetic compaction, cold isostatic pressing) and is generally determined by the compaction pressure [2].

Synthesis methods of highly transparent ceramics have been developed on the basis of calcination of nanopowders and compacts (Figure 2). The morphology, spectral and luminescence characteristics of laser ceramics, lifetimes of most important laser levels as well as the reasons of lasing absence under the doping of Nd:Y$_2$O$_3$ ceramic by Hf$^{4+}$ and Zr$^{4+}$ have been investigated [3].

![Figure 2. Photograph of ceramics produced in IEP UrB RAS](image)

It has been shown that substitution of Nd for Yb enabled the first highly efficient CW laser oscillation with a slope efficiency of $\eta=29\%$ (in collaboration with the Institute of Laser Physics SB RAS, ILP SB RAS) in a strongly disordered ceramic based on Y$_2$O$_3$ [4] and also with $\eta=51.2\%$ in a pulsed mode of operation (in collaboration with National Institute of Optics, INO CNR, Florence, Italy) [5]. Besides that, laser oscillations in Nd:YAG with $\eta=51.7\%$ (Figure 3, in collaboration with INO CNR) [6] and in Ho:YAG ceramics with $\eta=40\%$ (in collaboration with ILP SB RAS) [7] have been demonstrated.
Figure 3. Scheme of laser cavity and the dependences of the output power on the absorbed pump power for Nd:YAG ceramics fabricated by IEP UrB RAS (on the left) and by Konoshima Chemical Co. (on the right)

Research works on fabricating of magneto-optical ceramic for creation of devices based on Faraday effect such as optical circuits, mirrors, isolators, laser gyroscopes are carrying out. In recent years magneto-optical single crystals and glasses are synthesized using Ce, Pr, Dy and Tb oxides. Tb₂O₃ is the best among them and thus terbium gallium garnet (TGG) single crystals with the Verdet constant of 40 rad T⁻¹ m⁻¹ have found extensive application. Synthesis of magneto-optical ceramics has been described elsewhere [8,9]. We have succeeded in fabrication of highly transparent ceramics based on Tb₂O₃ (Figure 4). The obtained samples exhibit highest optical transmittance reported to date (82.5% at a wavelength of 1064 nm) and the Verdet constant of 120 rad T⁻¹ m⁻¹, which is three times higher than that of commercial TGG crystal. However the measured Verdet constant is inferior to the value obtained in [9].

Figure 4. Photograph and transmission spectra of ceramics based on Tb₂O₃

Synthesis technology of composite Nd:YAG/Cr,Ca:YAG ceramics for thin disk lasers and high power laser systems has been described elsewhere [10].
Diffusion bonding method for YAG ceramics has been developed. According to our investigations, the bonding interface has disappeared owing to recrystallization. Measurements of extinction coefficient have shown that extinction coefficient remained at the same level despite an increase in the thickness of bonded samples [11].

Ceramic Ce:YAG scintillator with good time-response characteristics and high light yield has been fabricated in collaboration with Ural Federal University [12].

Initial works on creating of new Fe$^{2+} \cdot $MgAl$_2$O$_4$ ceramics for active elements of mid-IR solid-state lasers, which are important for numerous civil and military applications, have been started. Figure 5 shows the photographs and transmission spectra of Fe$^{2+} \cdot $MgAl$_2$O$_4$ ceramics. It can be seen that with an increase in iron content the color of the samples changes from colorless to brown-red.

![Figure 5. Photographs and transmission spectra of ceramics with various iron content: a – 0.1 wt.%Fe$_2$O$_3$:MgAl$_2$O$_4$, b – 1 wt.%Fe$_2$O$_3$:MgAl$_2$O$_4$, c – 5 wt.%Fe$_2$O$_3$:MgAl$_2$O$_4$](image)

Transparency of samples is relatively low in the visible range, which is attributed to the presence of secondary phase (MgO)$_{0.9}$(FeO)$_{0.1}$ in ceramics with a content of several wt.% and crystallite sizes of ~120 nm. With an increase in wavelength the transparency increases and reaches 85.6% at a wavelength of 4 μm, which is close to the theoretical value of 86%. Further work in this direction will be associated with the elimination of secondary phase by selecting of nanopowders composition and sintering method.

The obtained results could serve as a basis for synthesis technology of various laser ceramics

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**References**
[1] Osipov V V, Kotov Yu A, Ivanov M G, Samatov O M, Lisenkov V V, Platonov V V, Murzakaev A M, Medvedev A I and Azarkevich E I 2006 Las. Phys. 16 116–25.
[2] Osipov V V, Platonov V V, Shitov V A and Maksimov R N 2017 Photonics 7 52–70.
[3] Osipov V V, Shitov V A, Maksimov R N and Solomonov V I 2015 *Opt. Mater.* **50** 65–70.
[4] Bagayev S N, Osipov V V, Shitov V A, Pestyakov E V, Kijko V S, Maksimov R N, Lukyashin K E, Orlov A N, Polyakov K V and Petrov V V 2012 *J. Eur. Ceram. Soc.* **32** 4257–62.
[5] Toci G, Pirri A, Patrizi B, Maksimov R N, Osipov V V, Shitov V A, Yurovskikh A S and Vannini M 2018 *Opt. Mater.* **83** 182–6.
[6] Osipov V V, Maksimov R N, Shitov V A, Lukyashin K E, Toci G, Vannini M, Ciofini M and Lapucci A 2017 *Opt. Mater.* **71** 45–9.
[7] Bagayev S N, Osipov V V, Vatnik S M, Shitov V A, Vedin I A, Platonov V V, Steinberg I Sh and Maksimov R N 2015 *Opt. Mater.* **50** 47–51.
[8] Snetkov I L, Permin D A, Balabanov S S and Palashov O V 2016 *Appl. Phys. Lett.* **108** 161905.
[9] Ikesue A, Aung Y L, Makikawa S and Yahagi A 2017 *Opt. Lett.* **42** 4399–401.
[10] Osipov V V, Shitov V A, Solomonov V I, Lukyashin K E, Spirina A V and Maksimov R N 2015 *Ceram. Int.* **41** 13277–80.
[11] Osipov V V, Lukyashin K E, Shitov V A and Maksimov R N 2016 *Mater. Lett.* **167** 81–4.
[12] Osipov V V *et al.* 2017 *Opt. Mater.* **71** 98–102.