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Feature issue introduction: optical ceramics

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Abstract: This feature offers 11 papers in the field of optical ceramics, and encompasses advances in optics, materials science, condensed matter, as well as physics and chemistry relevant to the development of new optical materials. Topics covered include material technologies in the field of polycrystalline ceramics, single crystals, and glass ceramics in the form of bulk and microstructured materials along with methods to fabricate the materials and a description of their optical properties pertinent for many applications.

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OCIS codes: (160.4760) Optical properties; (120.5700) Reflection; (120.5820) Scattering measurements; (160.5293) Photonic bandgap materials; (160.5298) Photonic crystals; (310.6188) Spectral properties; (160.0160) Materials; (220.4000) Microstructure fabrication; (160.5690) Rare-earth-doped materials; (300.6280) Spectroscopy, fluorescence and luminescence; (230.7370) Waveguides; (140.3390) Laser materials processing; (130.3120) Integrated optics devices; (160.1190) Anisotropic optical materials; (160.4236) Nanomaterials; (290.5930) Scintillation; (140.3380) Laser materials; (140.3580) Lasers, solid-state.

References and links

1. X. Qiao, S. Shimai, Y. Yang, S. Wang, and H. Kamiya, "Spontaneous gelcasting of translucent alumina ceramics," *Opt. Mater. Express* (to be published).
2. Y. Sato, J. Akiyama, and T. Taira, "Orientation control of micro-domains in anisotropic laser ceramics," *Opt. Mater. Express* **3**(6), 829–841 (2013).
3. Z. Seeley, N. Cherepy, and S. Payne, "Two-step sintering of Gd_{0.3}Lu_{1.6}Eu_{0.1}O₃ transparent ceramic scintillator," *Opt. Mater. Express* (to be published).
4. C. L. Hardin, Y. Koda, S. A. Basun, D. R. Evans, and J. E. Garay, "Transparent, luminescent terbium doped zirconia: development of optical-structural ceramics with integrated temperature measurement functionalities," *Opt. Mater. Express* **3**(6), 893–903 (2013).
5. J. Ueda, T. Shinoda, and S. Tanabe, "Photochromism and near-infrared persistent luminescence in Eu²⁺-Nd³⁺-co-doped CaAl₂O₄ ceramics," *Opt. Mater. Express* **3**(6), 787–793 (2013).
6. W. Kim, C. Baker, S. Bowman, C. Florea, G. Villalobos, B. Shaw, B. Sadowski, M. Hunt, I. Aggarwal, and J. Sanghera, "Laser oscillation from Ho³⁺ doped Lu₂O₃ ceramics," *Opt. Mater. Express* (to be published).
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10. S. N. Zhang, J. H. Huang, Y. J. Chen, X. H. Gong, Y. F. Lin, Z. D. Luo, and Y. D. Huang, "Site-selective excitation and emission of Eu³⁺-doped transparent glass ceramic containing Ca₅(PO₄)₃F nanocrystals," *Opt. Mater. Express* **3**(6), 868–874 (2013).
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The field of optical ceramics is very large and encompasses many aspects pertaining to polycrystalline material, single crystal, and glass-ceramic science. In what follows, we summarize the 11 contributions of this special feature according to these general categories.

There are seven papers in the polycrystalline materials category, which can be further subdivided into three groups that specifically describe process control, optical properties, and laser oscillation, respectively. The first group consists of three papers that highlight the

importance of controlling grain size and homogeneity through process optimization for making optically transparent ceramics of anisotropic materials. The first of these papers, by Qiao *et al.* [1], demonstrates fabrication of translucent alumina ceramics by a simple gelling system using a water-soluble co-polymer, acting as both a dispersant and a gelling agent. Gelation of the slurry occurred at room temperature in air without any other additives. Ceramics were obtained after drying and then sintering at 1850°C for 5 h in a vacuum furnace. Transmission of the resultant 1-mm-thick alumina ceramics was greater than 29% at 600 nm. Optimization of this process could provide a convenient way to make low-cost and fully transparent alumina ceramics for window related applications operating in hostile environments. In the second paper, Sato *et al.* [2] present a theoretical study on the orientation control of micro-domains in laser ceramics made from anisotropic crystals. They developed a distribution function of the crystal orientation in micro-domains and used detailed X-ray diffraction (XRD) analyses to demonstrate that preferential grain growth during the sintering process leads to improvement in the orientation distribution by the magnetic anisotropy due to *4f*-electrons of doped rare-earth ions. Optimization of those would provide a path forward for laser oscillation in ceramics made from anisotropic crystals that possess superior thermal, optical, and mechanical properties compared with their cubic counterparts. The third paper, by Seeley *et al.* [3], highlights the fabrication of transparent ceramic scintillators with composition $\text{Gd}_{0.3}\text{Lu}_{1.6}\text{Eu}_{0.1}\text{O}_3$ using a two-step sintering profile consisting of a fast ramp and short dwell followed by a long dwell at a lower temperature. A subsequent Hot Isostatic Press (HIP) step was used to achieve full density and transparency. Two-step sintering allowed full transparency to be achieved after HIPing at only 1525°C, compared with the significantly higher temperature of 1850°C required in the traditional sintering process. Their new process produces ceramics with submicron grain size compared with a grain size of hundreds of microns using the traditional process and emphasizes that their process allows densification to be decoupled from grain growth during their low temperature HIP step. Radioluminescence spectra show comparable results between samples with submicron grain size and those with 300 μm grains. Their approach has impact for all ceramics, as smaller grain size can lead to higher strength ceramics.

The second group of two papers describes optical properties of the rare-earth and transition metal-ion-doped ceramics. For example, Hardin *et al.* [4] present a method for the preparation of transparent Tb-doped zirconia ($\text{Tb}:\text{ZrO}_2$) ceramics that luminesce in the visible. The visible luminescence is temperature dependent, yielding samples that have integrated temperature sensing capabilities. They simultaneously react and densify ZrO_2 and Tb_4O_7 powder using current activated pressure assisted densification (CAPAD) which reduces sintering times and temperatures, leading to smaller grain size. The Tb dopant serves to both stabilize the tetragonal phase of zirconia and enables light emission. The $\text{Tb}:\text{ZrO}_2$ ceramics have an excellent combination of structural and optical properties; the toughness is comparable with yttria stabilized zirconia, and the transparency in the visible is high. The luminescent lifetimes are long and amenable to luminescent thermometry. The ceramics have promise as thermal barrier materials and high-strength windows with “built-in” temperature measurement capabilities. Ueda *et al.* [5] have discovered a photochromism phenomenon and near-infrared persistent luminescence of the Nd^{3+} : $^4\text{F}_{3/2} \rightarrow ^4\text{I}_{1/2}$ transition in $\text{CaAl}_2\text{O}_4:\text{Eu}^{2+}-\text{Nd}^{3+}$ ceramic. The sample color is changed from clear to pink after stopping UV irradiation due to the absorption of the color center. The persistent luminescence of Nd^{3+} is caused by the energy transfer from Eu^{2+} to Nd^{3+} . They report reflectance, photoluminescence, persistent luminescence spectra, and afterglow decay curves, and discuss the role of photo-oxidation, electron trapping, and de-trapping. Understanding these phenomena is important for enhancing capabilities of luminous paints, imaging plates for X-ray detection, and optical memories, etc.

The last group in the polycrystalline materials category contains two papers that highlight the first laser oscillation at 2 μm in bulk $\text{Ho}:\text{Lu}_2\text{O}_3$ ceramic and a novel waveguide

architecture written using a femtosecond (fs) laser in Yb:YAG ceramic, respectively. Kim *et al.* [6] report the first laser oscillation from 2% Ho³⁺:Lu₂O₃ hot pressed ceramic. Lu₂O₃ has one of the highest thermal conductivities of all the oxide hosts and therefore represents an excellent medium for high power lasers. Their high purity powders were synthesized by a co-precipitation method and then hot pressed into optical quality ceramic discs with transmission approaching the theoretical limit. The optical, spectral, and morphological properties are presented. Continuous wave lasing with an output power of 182 mW with a slope efficiency of 1% was observed at the eye-safe wavelength of 2.124 μm. Improvements in laser output power and efficiency are possible by eliminating trace amounts of phase/morphological impurities present in the ceramic. Jia *et al.* [7] demonstrate the fabrication of depressed double-cladding waveguides in Yb:YAG ceramics by using a novel fs laser inscription method. The double-cladding structures consist of tubular central structures with 30 μm diameter and concentric larger size tubular claddings with diameters of 100–200 μm. Continuous wave laser oscillation at a wavelength of 1030 nm has been realized at room temperature by pumping at 946 nm. The maximum output power obtained from these double-cladding waveguide lasers is ~80 mW with a slope efficiency of about 63%. Further improvements could lead to significantly higher power levels from these compact, integrated lasers.

On the second topic related to crystals, two papers describe the optical properties of inverse opal structures and those of a wide bandgap chalcopyrite, respectively. Lee *et al.* [8] describe titania inverse opal heterostructures demonstrating two distinctive photonic stopgaps fabricated by repetitive vertical self-assembly and atomic layer deposition (ALD). Angle resolved reflectance measurements of the inverse opal heterostructure are reported for the first time. A comparison with the spectra of the constituents shows that the stopgaps of the heterostructure obey the superposition principle and the angular dispersion of their stopgaps is well fitted with a modified Bragg's law at low incidence angles. Numerical simulations were used to predict the dominant features in the reflectance spectra. The total (specular and diffuse) transmission and reflectance measurements of the single inverse opals and the heterostructure reveal that the diffuse scattering could severely impair the photonic properties of the buried layers in the multi-stack photonic crystal (PhC) configurations. An ascending stacking is proposed as a means to improve the performance of the multi-layer coatings. Ho and Pan [9] have characterized the band edge and higher-lying interband transitions of CuAl(Se_{0.5}S_{0.5})₂ single crystals using thermorefectance (TR) measurements at room temperature. Single crystal needles were grown by a chemical vapor transport method. The TR measurement results showed three interband transitions denoted as E1, E2, and E3 detected near the band edge. Polarized thermorefectance (PTR) measurements showed that the E1 and E2 transition features are observed only with linearly polarized light along the $\epsilon_{||} < 111 >$ (needle axis) direction, while the E3 transition largely appears with the electrical field perpendicular to the needle axis. The E1 and E2 transitions may originate from the valence-band top, while the E3 transition is closely related to the valence-band splitting in the crystal. PTR measurements were carried out over a wide energy range of 2.5–6 eV to characterize the anisotropic properties of the electronic structure.

The third category on glass ceramics is represented by two papers that address the spectroscopy of rare-earth-ion-doped glass ceramics. Glass ceramics have drawn a lot of interest owing to their superior optical and mechanical properties compared with glasses. Zhang *et al.* [10] describe an Eu³⁺-doped transparent oxyfluoride glass ceramic containing Ca₅(PO₄)₃F nanocrystals prepared by melt quenching and subsequent thermal treatment. The transmittance of the glass ceramic with a thickness of 1.92 mm is up to 80.5% in the visible region. Site-selective excitation and emission spectra indicate that Eu³⁺ ions in the nanocrystals occupy two types of sites, A and B, with the same point symmetry C_s. The crystal field for Eu³⁺ ions at site A is more deformed and stronger and may be associated with a F⁻ ligand ion being replaced by an O²⁻ ion, in accordance with a charge compensation

scheme: $\text{Ca}^{2+} + \text{F}^- \rightarrow \text{Eu}^{3+} + \text{O}^{2-}$. As for the Eu^{3+} ions at site B, the possible charge compensation scheme is $3\text{Ca}^{2+} \rightarrow 2\text{Eu}^{3+} + \text{Vacancy}$. Furthermore, an energy transfer process from Eu^{3+} ions at site A to site B at low temperature was also discussed. Kamma *et al.* [11] describe a germano-tellurite based glass with 2.4% ErF_3 made by melt quenching. The Judd-Ofelt intensity parameters were estimated, and the radiative transition probabilities and lifetimes were also calculated. Nanocrystals were grown in the glass by heat treatment. Strong room temperature upconversion emissions were observed at 415, 540, 554, and 667 nm from an Er^{3+} -doped glass ceramic sample under 972 nm Ti-sapphire laser excitation. XRD measurements revealed the presence of 35 nm sized NaErF_4 crystallites in the glassy matrix. The concentration of nanocrystals was found to be low in the middle of the sample and higher close to the surface. Optimization of the processing could provide a reasonable path forward for making lasers for longer wavelengths than is currently possible in silicate glasses due to the lower phonon energy of the halide crystal matrix for the rare-earth ions.

The editors believe that the future of optical ceramics is strong and lies in its diversity and depth to address basic scientific problems within the context of applied practical challenges and limitations. We see great headway being made across the board in optical ceramics from passive optics to active optics, including rare-earth-doped lasers at eye-safer wavelengths. The editors note that the papers for this special issue represent authors from seven countries. We thank the authors for their outstanding work and encourage researchers to continue to develop exciting theoretical and applied research in the field of optical ceramics. We would like to express our gratitude to all authors and reviewers for their efforts in improving the manuscripts during the review process. We also thank David Hagan, Editor-in-Chief of *Optical Materials Express*, for his support and encouragement of this feature issue and the OSA journal staff for their excellent support during the review and production processes.