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Perovskite microwires generating red laser emission

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Abstract. Sources of stimulated coherent emission in all visible range are highly demanded for numerous applications in photonic and optoelectronic devices. In that regard, metal halide perovskite-based microlasers are established to be promising objects due to their low-threshold and lasing with high quality factor (more than 1000) at room temperature. However, there are still challenges in synthesis of stable and uniform crystals emitting red light. This work presents a novel sublimation synthesis method to produce mixed-anion perovskite CsPb(Br,I)₃ microwires that generate laser emission in red region of visible range. An increase in the iodine content in the structure contributes to a red-shift in photoluminescence from 530 nm to 680 nm. CsPb(Br,I)₃ microwires demonstrate multimode laser generation with threshold at 70 mJ/cm² and Q-factor near 1500. A uniform distribution of bromine and iodine ions in the structure is confirmed by energy dispersive x-ray analysis. Additionally, study of surface morphology by scanning electron microscopy showed core-shell like structure, which can improve long-term stability of obtained crystals. Our study reveals the sublimation as a promising synthesis method to produce low-threshold red microlasers that emit light at room temperature.

Keywords: halide perovskites, lasing, sublimation, red-shift, photoluminescence

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Конференционная статья

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Перовскитные нитевидные микрокристаллы, генерирующие лазерное излучение в красном диапазоне

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Аннотация. Источники стимулированного когерентного излучения во всем видимом диапазоне крайне востребованы для многочисленных применений в фотонных и оптоэлектронных устройствах. В этом отношении, микролазеры на основе металлгалогенидных перовскитов зарекомендовали себя как перспективные объекты благодаря низкопороговому и высокодобротному излучению (с показателем добротности больше 1000) при комнатной температуре. Однако до сих пор существуют проблемы с синтезом стабильных и однородных кристаллов, излучающих в красном диапазоне. В данной работе представлен новый метод сублимиционного синтеза для создания нитевидных смешанно-анионных перовскитных

микрокристаллов $\text{CsPb}(\text{Br},\text{I})_3$, генерирующих лазерное излучение в красной области видимого спектра. Увеличение содержания иода в структуре способствует сдвигу фотолюминесценции в красную область спектра от 530 нм до 680 нм. Микрокристаллы $\text{CsPb}(\text{Br},\text{I})_3$ демонстрируют многомодовую генерацию лазерного излучения с порогом 70 мДж/см² и добротностью около 1500. Равномерное распределение ионов брома и иода в структуре подтверждается энергодисперсионным рентгеновским анализом. Кроме того, исследование морфологии поверхности кристалла с помощью сканирующей электронной микроскопии показывает наличие структуры типа «ядро-оболочка», что может улучшить долговременную стабильность полученных кристаллов. Наше исследование показывает, что сублимация является перспективным методом синтеза для получения низкопороговых красных микролазеров, излучающих свет при комнатной температуре.

Ключевые слова: галогенные перовскиты, лазерная генерация, сублимация, смешение в красную область, фотолюминесценция

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Introduction

Perovskite crystals possess a number of outstanding properties, such as a low concentration of lattice defects, a high optical absorption coefficient, and a high luminescence quantum yield at room temperature [1]. In addition, microstructures can be fabricated in various resonant shapes, which enables generation of lasing emission in crystals [2]. Microscale sources of stimulated emission are highly demanded for application in various optoelectronic and photonic devices. For inorganic metal halide perovskite (MHP) crystals of the CsPbBr_3 composition the emission spectrum may be tuned by partial replacement of the halogen ions. The introduction of Cl^- or I^- ions instead of Br^- leads to a shift in the luminescence spectrum towards the blue or red region, respectively. However, due to the high mobility of iodine ions and its interaction with oxygen and water molecules in atmosphere, mixed crystals of this composition are highly unstable [3]. That results in formation of uneven distribution of Br^- -rich and I^- -rich domains under prolonged optical or electrical pump. Hence it leads to the shift of luminescence spectrum or the appearance of additional emission peak [4]. Such instability of spectra is especially pronounced for mixed-iodine crystals that are synthesized at rather low temperatures (below 100 °C) through anion exchange where the source of halogen is gas [5] or another pure iodine structure [6] as the migration process mainly happens at the surface level. In this work, the new sublimation method was employed for creating microstructures that exhibit narrow and smooth photoluminescence peak at room temperature in red range. Here, the crystals are synthesized at higher temperatures which facilitates the I^- ions uniform diffusion within all crystal volume and the additional shell-like layer of PbI_2 protects perovskite from the external environment.

Materials and Methods

Two types of solutions were prepared to produce polycrystalline perovskite source-substrates. They were obtained by dissolving CsBr , PbBr_2 and PbI_2 powders in dimethyl sulfoxide to produce pure solutions with 0.3 M for CsPbBr_3 and 0.075 M for PbI_2 . At the beginning, 2 types of sources for subsequent synthesis of microwires (MWs) by sublimation technique were prepared. Firstly, the CsPbBr_3 solution was drop-cast on a glass substrate and placed in a petri dish in isopropyl vapor at 100 °C for 10 minutes to form CsPbBr_3 crystals. Secondly, the same protocol was repeated

for the PbI_2 solution. In this work, glass substrates coated with $\text{CsPbBr}_3/\text{PbI}_2$ microcrystals were used as the source substrate, and a mica substrate was used as the receiver substrate. Source substrate was placed on a high-temperature plate and a receiver substrate was fixed on top of it at a 2.5 cm distance. Initially, the CsPbBr_3 substrate was used as a source to create microwires on the mica, and later lead iodide was evaporated onto the resulting microwires, thereby allowing the formation of mixed $\text{CsPb}(\text{Br},\text{I})_3$ microstructures. After that the samples were cooled down to room temperature.

The surface morphology and elemental analysis of obtained mixed-anion perovskite microwires were studied by a scanning electron microscopy (SEM) method and energy-dispersive X-ray (EDX) analysis (FEI Quanta Inspect). The bright-field and luminescent images of $\text{CsPb}(\text{Br},\text{I})_3$ MWs with different iodide concentration were obtained by C.Z.A Imager 2 microscope (Carl Zeiss). The photoluminescence spectra were measured on a QE-Pro spectrometer (Ocean Optics) coupled to microscope with optical fiber. Lasing properties of single microwire were studied under femtosecond laser excitation (Antaus, Avesta) with pulse width $\tau = 150$ fs at a wavelength of 515 nm and 10 kHz repetition rate. The laser beam was focused onto the sample surface at normal incidence by a microscope objective (50 \times , NA = 0.42) aligned to provide uniform irradiation of the microwires (Gaussian distribution with an FWHM of ≈ 20 μm). The signal was separated from pump via long-pass dichroic mirror (cut-off wavelength of 550 nm) and registered on a spectrometer (Andor Kymera 328i, Oxford instruments) equipped with a cooled electron multiplying CCD camera. All measurements were performed at normal conditions and reproduced several times.

Results and Discussion

Arrays of synthesized microwires were visualized by bright-field images from optical microscope. The produced crystals varied in cross-section size and total length (5 to 20 microns) (Fig. 1, *a*). In addition, the fluorescent images show homogeneous luminescence of crystals (Fig. 1, *b*). The EDX mapping analysis revealed uniform distribution of elements within the crystal which indicates its structural stability (Fig. 1, *c*). The photoluminescence spectrum of the MW was 577 nm, which corresponded to 16% of iodine concentration in the structure. Notably, it was discovered that iodine deposition creates a so-called ‘coat’ over the obtained CsPbBr_3 microwires,

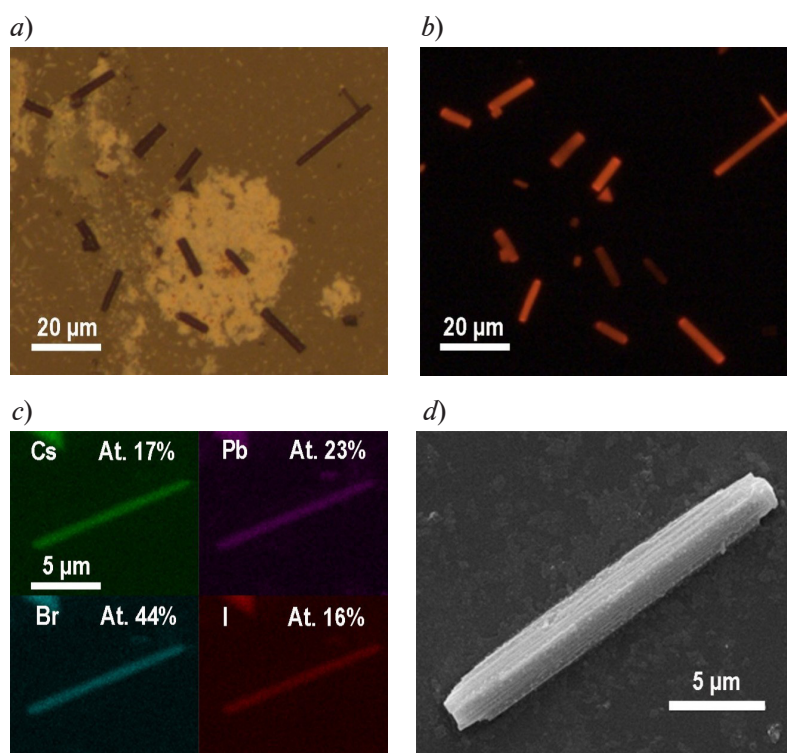


Fig. 1. Characterization of mixed-anion $\text{CsPb}(\text{Br},\text{I})_3$ microwires: bright-field (*a*) and fluorescent (*b*) images of obtained microwires; EDX mapping of single MW (*c*); SEM-image of a single MW (*d*)

as clearly seen in the SEM image (Fig. 1, *d*). We suggest that the produced mixed MWs possess a core-shell-like structure, where the iodine-based ‘shell’ protects perovskite ‘core’ from oxygen and moisture, thereby making them more stable over time.

A comparison of optical properties of obtained mixed-anion microwires was made by measuring photoluminescence (PL) spectra (Fig. 2, *a*). For the reference microwire without iodine, the photoluminescence peak was 530 nm. A subsequent increase of the iodine concentration in the structures allows one to observe a red shift in the photoluminescence spectra (see Fig. 2, *b* for a visual representation). At the point of maximum iodine concentration, the photoluminescence peak is located at a wavelength of 680 nm. The difference in the full width at half maximum of the measured photoluminescence spectra can be explained by the difference in the lateral dimensions of the mixed perovskite microwires.

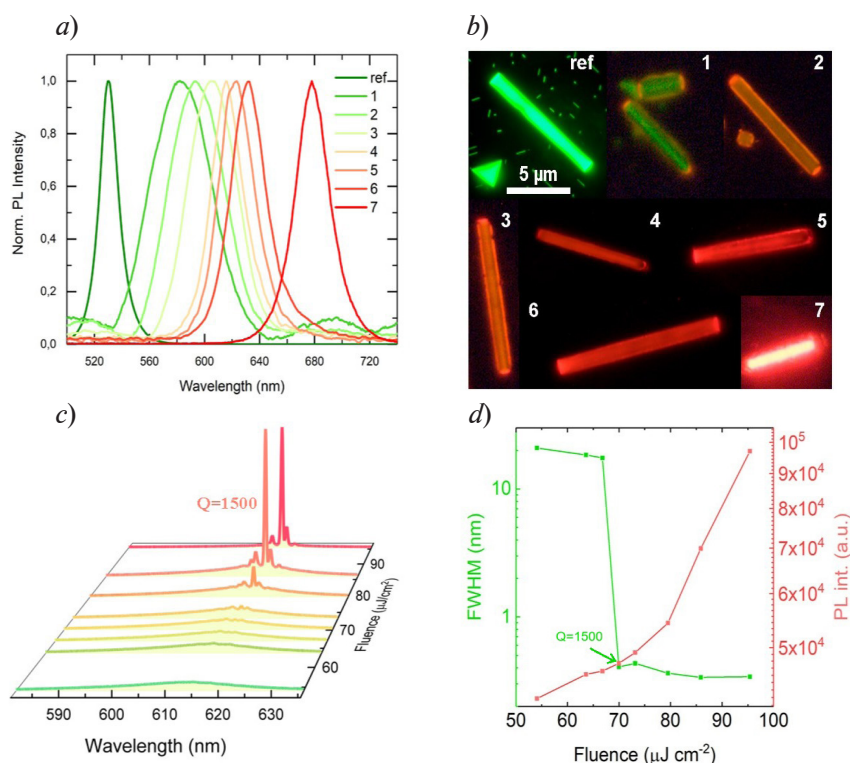


Fig. 2. Optical characterization of $\text{CsPb}(\text{Br},\text{I})_3$ microwires: photoluminescence spectra of isolated microwires 1–7 found at different locations on the substrate (*a*); fluorescent images of microwires 1–7, respectively (*b*); photoluminescence spectra for $\text{CsPb}(\text{Br},\text{I})_3$ microwire at different intensities below and after the lasing threshold (*c*); intensity and full-width-half-maximum (FWHM) of emission spectrum of the microwire at the lasing wavelength (*d*)

To demonstrate the lasing properties of MWs, they were pumped by a femtosecond laser emission. Fig. 2, *c* reveals the spectral evolution of PL with the increase of the laser pump fluence for a single mixed-anion perovskite microwire with lateral size 15 μm . It worth noting that there was no PL spectral split contributed to iodine migration, which confirms the stability of luminescence in crystals. The observed behavior demonstrates all basic lasing features: clear lasing threshold at fluence = 70 $\mu\text{J}/\text{cm}^2$ and its sharp spectral narrowing by two orders of magnitude at the red wing ($\lambda = 620\text{--}625$ nm) of excitonic PL spectra as shown in Fig. 2, *d*. Quality factor (Q) of the most intense lasing mode was estimated to be 1500 and calculated as $Q = \lambda/\Delta\lambda$, where λ is the mode center wavelength and $\Delta\lambda$ is the mode width.

Conclusion

In this work we presented a novel synthesis method of mixed-anion $\text{CsPb}(\text{Br},\text{I})_3$ perovskite microwires which demonstrated laser generation in red region of visible range. The obtained perovskite microwires showed a stable peak of photoluminescence without any shifts throughout



all experiments at room temperature. Lasing threshold was estimated to be $70 \mu\text{J}/\text{cm}^2$ with quality factor near 1500. Produced structures can be used in optoelectronic and photonic devices, which require stable photoluminescence and laser emission.

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