A liquid phase anion-exchange approach to high-quality all-inorganic halide perovskite micro- and nanowires

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ABSTRACT

All-inorganic halide perovskite nanowires (NWs) are promising materials due to they have broad application prospects in the field of optoelectronics, with mixed-halide perovskite nanowires can change the optoelectronic properties by adjusting the halide ratio. Here, we experimentally investigated the two-process governed anion-exchange reaction in single-crystalline CsPbX₃ micro- and nanowires. The critical parameters affecting the outcome of the reaction are identified as the reaction temperature, reaction time, and precursor concentrations. Upon examining the photoluminescence and morphology of the NWs, high-quality NWs were obtained by optimizing these critical parameters. The bandgap of the NWs can be tuned over the entire visible spectra (430–700 nm). In addition, photodetectors incorporating single NWs were fabricated, which demonstrated excellent responsivity under illumination. Our results expand the validity of liquid-phase anion exchange to the microscale, and lay the basis for liquid-processed optoelectronics and displays.

Introduction

As an emerging new class of semiconductor material, halide perovskites have become a promising candidate for applications in solar cells [1–7], light-

emitting diodes [8–10], and photodetectors [11–18]. In recent years, low-dimensional halide perovskite nanostructures including quantum dots [19–21], nanowires (NWs) [22–27], and ultra-thin nanosheets [28] have been widely reported, leading to new opportunities in a variety of optoelectronic devices

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[29]. Compared to conventional semiconductors, halide perovskites possess many unique advantages including low-cost solution processability, high defect tolerance, and tunable optical properties. Particularly, the “soft” lattice of halide perovskites, which originates from the weak ionic bonding in this class of semiconductors, results in a highly dynamic lattice ideal for bandgap engineering [30]. Anion exchange has been demonstrated to be a simple and effective approach for bandgap engineering in halide perovskite nanostructures [31]. The exchange reaction can occur at the solid–gas, solid–liquid, and liquid–liquid interface [32]. In nanometer-sized quantum dots [31] and quantum wires [25], it has been shown that the anion-exchange reaction can happen at a strikingly high speed of 0.3 s. Comparatively, in larger single-crystalline structures, the anion exchange is much slower, as it generally involves two consecutive processes: the exchange reaction at the surface of the crystal, followed by the interdiffusion of halogen ions inside the crystal. In an ideal anion exchange, these two dynamic processes must balance each other. However, to our best knowledge, there is no comprehensive research of such anion exchange in the micron and sub-micron regimes. Here, we experimentally investigated the two-process governed anion-exchange reaction in single-crystalline CsPbX_3 micro- and nanowires. The critical parameters affecting the outcome of the reaction are identified as the reaction temperature, reaction time, and precursor concentrations. Upon examining the photoluminescence and morphology of the NWs, high-quality NWs were obtained by optimizing these critical parameters. The bandgap of the NWs can be tuned over the entire visible spectra (430–700 nm). In addition, photodetectors incorporating single NWs were fabricated, which demonstrated excellent responsivity under illumination. Our results expand the validity of liquid-phase anion exchange to the microscale, and lay the basis for liquid-processed optoelectronics and displays.

Materials and methods

Materials

1-Octadecene (ODE, 90%, Alfa Aesar), oleic acid (OA, 90%, Alfa Aesar), oleylamine (OAm, 80–90%, Acros Organics), n-Hexane (98%, Aladdin), chlorobenzene

(99.8%, Sigma-Aldrich), N, N-Dimethylformamide (DMF, 99.8%, Aladdin) Isopropyl alcohol ($\geq 99.7\%$, Kermel), PbBr_2 (99.999%, Macklin), PbI_2 (99.9985%, Alfa Aesar), PbCl_2 (99.999%, Aldrich), CsBr (99.999%, Aldrich). All chemicals were used without further purification.

Synthesis of CsPbBr_3 NWs with green light emission

The FTO glass substrate was placed in deionized water, acetone, and isopropanol for ultrasonic cleaning for ten minutes. At room temperature, CsBr and PbBr_2 were dissolved in DMF at a molar ratio of 1:1 to obtain a mixed solution. The FTO glass substrate was placed in a beaker containing an anti-solvent in advance, where the FTO glass substrate doesn't contact the anti-solvent liquid. The anti-solvent volatilizes at room temperature, forming a vapor atmosphere in the beaker. The mixed solution was dropped on the FTO glass substrate, and then sealed the beaker with a plastic wrap, and incubated at room temperature in a closed anti-solvent vapor environment for 2–12 h. The CsPbBr_3 NWs were cleaned with isopropanol and blow-dry with a nitrogen gun. The anti-solvent includes isopropanol and chlorobenzene.

Anion Exchange of CsPbBr_3 nanowires

In a glove box filled with nitrogen, PbX_2 ($\text{X} = \text{Cl}, \text{I}$) was dissolved in 1-Octadecene, where the concentration of PbX_2 is 2–4 mg/ml. Then add a small number of organic additives oleic acid and oleic acid amine (0.1–0.5 ml), using a water bath method, performed magnetic stirring at a temperature of 80–100 °C, and used it as an ion source after PbX_2 was completely dissolved. The CsPbBr_3 NWs were immersed in the ion source and reacted for 1–24 h in an environment with a temperature of 25–50 °C to obtain perovskite nanowire crystals with different halides components.

Characterization

X-ray powder diffraction (XRD) was performed using a SmartLab 9kw03030502 X-ray powder diffractometer with $\text{Cu K}\alpha$ radiation to characterize the perovskite nanocrystal structure. The scanning electron microscope (SEM) images were taken with an

FEI NOVA Nano SEM. The energy-dispersive X-ray spectroscopy (EDS) elemental mapping images were recorded using an FEI NOVA Nano SEM operated at 20 kV. The steady-state PL emission spectra were recorded on a SpectraPro HRS-500 fluorescence spectrophotometer with a U-LH100HG lamp coupled to a monochromator. The PL image was performed using Digital Camera DP800 by an OLYMPUS BX53F microscope. The measurement of TRPL uses the DeltaFlex03190728 ultrafast fluorescence lifetime spectrometer of HORIBA Company. All spectral measurements were performed at room temperature.

Results and discussion

To perform the anion-exchange reactions, we firstly synthesized CsPbBr_3 NWs as the templates. The NWs were grown in a nitrogen-filled glovebox via a one-step solution method. First, PbBr_2 (0.1 mM) and CsBr (0.1 mM) precursors were dissolved, respectively, in dimethylformamide (DMF) to form a mixed solution. The FTO glass substrate was placed in a beaker containing an anti-solvent in advance, where the FTO glass substrate doesn't contact the anti-solvent liquid. The mixed solution was dropped on the FTO glass substrate at room temperature for 2–12 h to obtain NWs. The as-grown NWs were examined by a scanning electron microscope (SEM). As shown in Fig. 1a, the growth substrate was uniformly covered by NWs with diameters ranging from 100 nm to 2 μm and length up to tens of microns. Each NW exhibits a smooth surface and a square-shaped cross-section. To confirm the lattice structure of these NWs, X-ray diffraction (XRD) was conducted (Fig. 1b), and the obtained diffraction pattern coincides with the standard XRD patterns of monoclinic CsPbBr_3 (PDF-18-0364). The strong diffraction peak at 30.6° corresponds to the (200) facet of CsPbBr_3 . As most NWs are horizontally deposited on the substrate, the axial direction of the NWs is along the (200) direction. Subsequently, the photoluminescence (PL) spectra of single CsPbBr_3 NWs were measured under a microscope. As shown in Fig. 1a, d, sharp PL emission peak was detected at 527 nm with a full width at the half-maximum (FWHM) of 17.9 nm. The corresponding fluorescence image (Fig. 1c) shows uniform green light emission along the NW, with enhanced emission at both end facets of the NW due to optical resonance modes in the NW cavity [33–36].

Figure 1 Characterizations of CsPbBr_3 NWs. **a** SEM image of NW growth substrate, **b** XRD pattern of NWs, **c** PL image of a single CsPbBr_3 NW, and **d** PL spectra of a typical CsPbBr_3 NW.

Overall, the above characterizations indicate that as-grown CsPbBr_3 NWs possess high crystal quality and excellent photoluminescence performance, and can be served as good templates for the following anion-exchange reactions. Next, we converted the CsPbBr_3 NWs into alloy $\text{CsPb}(\text{Br}/\text{X})_3$ ($\text{X} = \text{Cl}, \text{I}$) NWs. The anion-exchange solution was prepared by dissolving PbX_2 into a 1-Octadecene solution. The NW-containing FTO substrates were dipped into the exchange solution under a nitrogen atmosphere. The degree of the anion exchange, namely the Br/X ratio, can be adjusted by controlling the reaction temperature, reaction time, and the concentration of the exchange solution. In an optimized reaction, the reaction time, temperature, and solution concentration are 1–24 h, 25–50 $^\circ\text{C}$, and 2–4 mg/ml, respectively. The quality of the acquired alloy NWs was examined by SEM and PL measurements. As shown in Fig. 2a, b, the alloy NWs maintain a damage-free smooth surface after anion exchange, and the color of PL emission evolves continuously from green to blue and green to red in $\text{CsPb}(\text{Br}/\text{I})_3$ and $\text{CsPb}(\text{Br}/\text{Cl})_3$ NWs, respectively (Fig. 2a, b inset). According to the formula $E_g = 2.34 - 0.27x - 0.29x^2$ summarized in the literature [37], combined with the PL peak value of the $\text{CsPb}(\text{Br}/\text{I})_3$ NWs, the ratio of I elements in the nanowire can be calculated, which has been marked in Fig. 2a. Where E_g is the bandgap of the NWs, and x

Figure 2 SEM image of **a** CsPb(Br/I)₃ and **b** CsPb(Br/Cl)₃ NWs. The insets show the evolution of the emission color. **c**, **d** EDS mapping for CsPb(Br/I)₃ **c** and CsPb(Br/Cl)₃ **d** single NWs.

is the ratio of the I element. Using the same method, the proportion of Cl element in CsPb(Br/Cl)₃ NWs can be calculated [38]. The PL image of individual alloy NWs is uniform, and no core-shell structures were observed, indicating the complete formation of uniform alloy composition. To further support this argument, we applied the energy dispersive spectrometer (EDS) to map the spatial distribution of each element (Fig. 2d, e). The EDS results clearly show evenly distributed chlorine and iodine ions.

In addition, we studied the PL spectra of the alloy NWs in detail. As shown in Fig. 3a, the emission peak gradually blue-shifted from 527 (pure CsPbBr₃) to 430 nm in CsPb(Br/Cl)₃ NWs, and red-shifted up to 700 nm in CsPb(Br/I)₃ NWs. Compared to pure CsPbCl₃ (422 nm) and CsPbI₃ (740 nm) [39], the liquid anion-exchange method can nearly achieve complete composition conversion without apparent damage to the NWs. We also examined the uniformity of the alloy NWs by measuring the point-by-point PL at various locations on a single NW. As shown in Fig. 3c, the four selected points from the same NWs demonstrate the exact PL spectra, consistent with the EDS results. The FWHMs of each alloy NWs were calculated and plotted in Fig. 3b. Overall, the FWHMs of CsPb(Br/Cl)₃ NWs have no significant change, while the FWHMs of CsPb(Br/I)₃ NWs exhibit a slight increase from 17.9 to 33.4 nm. The lattice mismatch between CsPbCl₃ and CsPbBr₃ is 0.04, and 0.18 between CsPbBr₃ and CsPbI₃.

Therefore, the larger lattice mismatch may result in higher defect density in CsPb(Br/I)₃ NWs. To further verify this, we performed time-resolved photoluminescence (TRPL) on the alloy NWs (Fig. 3d). The TRPL results were fitted individually to a biexponential decay function (Fig. 3d), with carrier lifetimes for CsPbBr₃ and alloy NWs shown in Fig. 3e and Table S1. The average carrier lifetime of CsPb(Br/I)₃ NWs is 2.57 ns higher than the 1.24 ns of CsPb(Br/Cl)₃ NWs. This is due to the trap effect caused by the higher defect density in CsPb(Br/I)₃ NWs.

We then studied the effect of temperature, reaction time, and exchange solution concentration on the quality of the alloy NWs. As discussed above, the whole anion exchange contains two processes: (1) the exchange reaction at the NW surface and (2) the ion interdiffusion inside the NW (Fig. 4a). The former process strongly depends on the concentration of halogen ions, and the latter follows the effective vacancy-assisted diffusion mechanism. Due to the low ion mobility in bulk perovskites, the speed of ion interdiffusion is often much slower than that of the surface reaction. The experimental conditions have a profound effect on these two processes. First of all, with the extension of the exchange time, the two processes will naturally deepen gradually, as shown in Fig. 4b, Fig. S1, and Fig. S2. The PL spectrum of the CsPb(Br/I)₃ NWs has a very obvious redshift, and the PL spectrum of the CsPb(Br/Cl)₃ NWs has a very obvious blueshift. Increasing the temperature from 25 to 50 °C under the same experimental conditions can speed up the exchange reaction process (Fig. 4c). Temperature can accelerate the free movement of ions. Therefore, these two processes of anion exchange will be greatly promoted. In the case of other experimental conditions unchanged, increasing the concentration from 2 to 4 mg/ml will also speed up the exchange reaction (Fig. 4d). But compared to temperature, the effect of concentration is significantly smaller. This is because the increase in concentration only affects the process of halogen ions entering the surface, and it is difficult to affect the process of ions inside the NW. In the experimental method, the quantum wires need to be obtained by the centrifugal method [25], but the experimental method in this article is more concise. Just the NW-loaded substrate was immersed in the precursor solution and took it out after the reaction. In addition, the CsPbBr₃ NWs (100 nm–2 μm) used for the anion exchange performed here have a larger size in width

Figure 3 **a** PL spectra of alloy NWs. **b** FWHM of each curve in **(a)**. **c** PL spectra at various points in three alloy NWs. **d** and **e** Carrier lifetimes of alloy NWs as a function of wavelength.

than the anion exchange of the CsPbBr₃ quantum wires (1–10 nm) [40]. Therefore, the experiment can perform detailed analysis and research on a single nanowire on the substrate. Anion exchange requires two steps in micro- and nanowires, with a more complex exchange mechanism. Our experiment studied the exchange mechanism in detail.

In addition, we fabricated single NW photodetectors based on the CsPbBr₃ and alloy NWs. The NWs were transferred to pre-patterned gold electrodes by a micromanipulator. The single NW photodetector with a structure of Au/NW/Au is constructed and shown in Fig. 5a. The typical device channel length is 20 μm (Fig. 5b inset). The *I*–*V* curves of single NW devices were measured in dark and illumination under a 405 nm light-emitting diode at 2.5 mW/cm². As shown in Fig. 5b, the CsPbBr₃ single NW

photodetector demonstrates strong photoresponse, with photoresponsivity of 8.7×10^7 nA/mW at 5 V. We subsequently measured the photoresponse of alloy NWs (Fig. 5c). The alloy NW devices exhibit photocurrent similar to the CsPbBr₃ NWs with decreased magnitude, indicating loss of photocarriers through recombination. The CsPb(Br/Cl)₃ NW shows the smallest photoresponse, consistent with its shortest lifetime measured from the TRPL measurement. In order to quantitatively evaluate the response time, the enlarged temporal photoresponse results are displayed in Fig. 5d and Fig. S3. Rise and decay times are calculated to be 82.35 ms and 40.76 ms, respectively, which are defined as the time interval from 10 to 90% of saturated photocurrent. Although it is a single nanowire photodetector, it still

Figure 4 **a** Schematic diagram of the two-step anion-exchange process. **b–d** Evolution of PL peaks as a function of **b** time, **c** temperature, and **d** PbI_2 concentration.

Figure 5 **a** Schematic diagram of a single nanowire photodetector. **b** I – V curves of a single CsPbBr_3 NW in dark and under illumination. The inset shows an optical image of the device. The scale bar is 10 μm . **c** The I – V curves of alloy NWs under illumination. **d** I – t curve of a single CsPbBr_3 NW photodetector at 5 V under 405 nm.

has a good light response compared to previous photodetectors [41, 42].

Conclusions

In conclusion, we comprehensively studied the solution-phase anion exchange of halide perovskite NWs on the sub-micron to micrometer scale. The

two-step anion-exchange process can convert CsPbBr₃ NWs to a wide range of alloy NWs with a bandgap covering the entire visible spectra. We found that balancing the speed of the two processes is critical to achieving high-quality alloy NWs. This study shines a light on solution-processed optoelectronics and photovoltaics, and broadens the application prospective of halide perovskites.

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Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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