Metal seeding growth of three-dimensional perovskite nanowire forests for high-performance stretchable photodetectors

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ABSTRACT

Metal halide perovskites have been ubiquitously utilized in both rigid and flexible optoelectronics with high performance. However, developing perovskites-based stretchable optoelectronics is highly challenging because perovskites are intrinsically not stretchable unlike some organic semiconductors. Here, for the first time, we devised a novel metal seeding growth method to obtain three-dimensional (3D) perovskite nanowire forests (NWFs) with excellent mechanical stretchability, akin to the seeding growth of grass in nature. Benefiting from their intrinsic stretchability, perovskite NWFs-based stretchable photodetectors have been demonstrated with excellent photodetection performance. Under 0% strain, the device dark current is as low as 0.18 nA cm⁻² at V bias, contributing to an ultrahigh light-to-dark current ratio (I light/I dark) of 1.1 × 10⁵ under 5.5 mW cm⁻² illumination. The device also exhibits a high responsivity of 455.3 mA W⁻¹ and specific detectivity of 8.69 × 10¹² Jones. Meanwhile, under 80% strain conditions, the device have shown negligible performance drop, especially in the aspect of I light/I dark. Our work reports a novel growth process that leads to intriguing 3D perovskite nanostructures with unique mechanical flexibility and stretchability, in addition to high optoelectronic sensing performance. These appealing properties can find applications in high-performance stretchable and wearable optoelectronics in the future.
1. Introduction

High-performance stretchable electronics and optoelectronics, such as photodetectors, chemical and pressure sensors, light emitting diodes, energy harvesting/storage devices, etc., have garnered enormous research interests because of their great potential in wearable electronics, implantable biomimetic devices, smart clothes, and artificial skins [1–10]. Typically, there are mainly two approaches to fabricate stretchable electronic devices: integrating flexible materials with substrate geometric engineering, and embedding intrinsically stretchable materials into elastomeric substrates [11,12]. For the first approach, the geometrically engineered structure can be wavy, buckled, wrinkled, serpentine, island, or net configurations. For example, Kaltenbrunner et al. laminated an ultra-lightweight flexible electronic foil onto a pre-stretched elastomeric substrate [13]. After releasing the pre-stretched substrate, wrinkles were formed on the electronic foil, which enabled good stretchability of the device in the direction of the initial deformation of the substrate. In the second strategy, intrinsically stretchable electrodes, dielectrics, and semiconductors are preliminarily obtained and integrated into elastomeric substrate. For instance, Lee et al. embedded intrinsically stretchable ZnO nanowires (NWs) and Ag NWs in polydimethylsiloxane (PDMS)-a well-known elastomer- for stretchable photodetectors, which could maintain their functionalities even under 100% strain [14]. This strategy shows significant advantages over the geometric engineering approach, such as simple fabrication, low cost, high yield, and multi-directional stretching, etc., making it more attractive for the practical stretchable electronics and optoelectronics.

Metal halide perovskites, hereafter referred to as perovskites, have been demonstrated with great potentials in next-generation electronics and optoelectronics with high performance [15–21]. Especially, benefiting from the long carrier diffusion length (micrometers level in single crystals) [22], high carrier mobility (order of 10 cm² V⁻¹ s⁻¹) [23] and large light absorption coefficient (order of 10⁴ cm⁻¹) [24], perovskite photodetectors (PDs) have achieved remarkable accomplishment in device performance [20,25–28]. For example, a record responsivity value of 2.6 × 10⁶ A W⁻¹ [26] and specific detectivity value of 7 × 10¹⁵ Jones [27] have been reported in state-of-the-art rigid perovskite PDs, which are much higher than that of many traditional semiconductors-based PDs, such as Si, GaAs, and organic semiconductors [29–31]. However, very few stretchable perovskite PDs have been reported before, which seriously hampers their applications in stretchable optoelectronics [32–35]. Even though a pre-strained substrate has been used to obtain perovskite microwires-based PDs with certain stretchability, [33] fabricating intrinsically stretchable perovskite materials is still very challenging to obtain high-performance stretchable perovskite PDs. Three-dimensional (3D) NWs networks have been demonstrated with good intrinsic stretchability [36]. However, the growth of 3D perovskite NWs are mostly limited within rigid templates with both solution and vapor methods [37,38], which limits their applications in stretchable optoelectronic devices.

In this work, we devise a metal seeding growth (MSG) method for fabricating 3D vertical perovskite NW forests (NWFs) with excellent mechanical stretchability, and further utilize them in high-performance stretchable PDs. The MSG is based on a vapor-solid-solid reaction (VSSR) on SiO₂-covered porous alumina membranes (PAMs) with Pb seed embedded inside. During the NWFs growth, the precursor vapor, SiO₂ @PAMs, and Pb metal are “nutrients”, “soils”, and “seeds”, respectively, akin to the seeding growth of grass in nature. The NWFs have an average NW diameter of ~ 200 nm and forest height of ~ 50 µm, with high NWs density. After embedding them into elastomeric PDMS and employing Ag NWs as stretchable electrodes, an intrinsically stretchable perovskite PD is obtained with excellent device performance and mechanical stretchability. The device possesses an ultralow dark current of 8.57 nA cm⁻² at – 15 V bias, and 0.18 nA cm⁻² at – 2 V bias, contributing to an ultrahigh light-to-dark current ratio (I_{light}/I_{dark}) of 1.1 × 10⁸ under 5.5 mW cm⁻² illumination. Meanwhile, a high responsivity of 455.3 mA W⁻¹, a high specific detectivity of 8.69 × 10¹² Jones, and a low detection limit of 0.84 µW cm⁻² have been demonstrated. Most importantly, the device can maintain almost 90% light current and response speed even under 80% strain. To the best of our knowledge, this is the first demonstration of intrinsically stretchable perovskite PDs without substrate engineering. Our findings open a new vista for perovskite nanomaterials in high-performance stretchable electronics and optoelectronics.

2. Results and discussions

The MSG process and device fabrication are schematically illustrated in Fig. 1 (a). Different from what have been demonstrated before that PAM channels work as templates for perovskite NWs growth with

![Fig. 1. Schematic demonstration of (a) the MSG and stretchable device fabrication processes, and (b) the seeding growth of grass in nature.](image-url)
overgrown thin film on the surface (Fig. 1(a2), and Supplementary Fig. S1), SiO$_2$-covered PAMs with electro-deposited Pb metal in the channels (SiO$_2$@Pb@PAMs) are utilized here to grow the perovskite NWFs (Fig. 1(a3) and (a4)). Different from well-known vapor-liquid-solid (VLS) growth of conventional inorganic NWs, VSSR is a self-catalytic growth method, where the metal Pb serves as a reactant instead of a catalyst. The totally different growth mechanism leads to an overall reaction as followings:

$$3\text{CH}_3\text{NH}_3\text{I} + \text{Pb} \rightarrow \text{CH}_3\text{NH}_3\text{PbI}_3 + 2\text{CH}_3\text{NH}_2 + \text{H}_2$$

In such a VSSR process, SiO$_2$@PAMs and Pb metal are “soils” and “seeds”, respectively, akin to the seeding growth of grass in nature (Fig. 1(b)). Before embedding the obtained perovskite NWFs into high elastomeric PDMS for the stretchable PD device, a thin layer of Parylene C is conformably coated on the perovskite NWs to promote the PDMS curing (Supplementary Fig. S2), as schematically demonstrated in Fig. 1(a5) and (a6). After applying the stretchable Ag NWs electrode, a stretchable device is obtained and schematically shown in Fig. 1(a7).

The cross-sectional scanning electron microscope (SEM) image in Fig. 2(a) and energy-dispersive X-ray spectroscopy (EDS) mapping in Fig. 2(b) clearly show the SiO$_2$@Pb@PAMs structure, where Pb seed is fully embedded in SiO$_2$@PAMs template. When placing it in methylammonium iodide (MAI) vapor environment under 180 °C, the MAI will penetrate through SiO$_2$ layer and react with Pb seeds to produce MAPbI$_3$. Then the produced MAPbI$_3$ will grow along the grain boundary of SiO$_2$ layer (Fig. 2(c)) and form vertical NWs. Fig. 2(d) is a cross-sectional SEM image of the grown NWFs, showing a height of ~50 µm with very high NWs density. Even though similar seeding growth technique has been reported for growing perovskites thin films [44–47] and inorganic single crystals [48,49], to the best of our knowledge, it is the first time that vertical NWFs are fabricated with the MSG method. Fig. 2(e) and (f) show the top-view SEM images of the NWFs in low and high magnifications, respectively. It can be seen that those NWs have an average diameter of ~200 nm with rectangular ends, indicating a cubic perovskite phase. The EDS mapping results shown in Fig. 2(g)-(i) reveal uniform perovskite composition distribution in the NWFs. Element analysis with a single NW is shown in Supplementary Fig. S3, where a larger halide/Pb ratio can be found because of the presence of iodine interstitials.

Meanwhile, it is interesting that the obtained MAPbI$_3$ morphology can be controlled by both SiO$_2$ thickness and deposition rate, as shown in Supplementary Fig. S1 and S4. If there is no extra SiO$_2$ layer on PAMs, MAPbI$_3$ thin film is obtained because MAPbI$_3$ NWs from the neighboring channels will merge with each other on the surface (Supplementary Fig. S1). After adding a thin layer (50 nm) of SiO$_2$, both crystals and short NWs can be observed after the VSSR growth (Supplementary Fig. S4).

![Fig. 2. MAPbI$_3$ NWFs growth and characterization. (a-c) Pb NWs precursor in SiO$_2$/PAMs: (a) cross-sectional SEM image, (b) EDS mapping, and (c) top-view SEM image. (d-i) grown MAPbI$_3$ NWFs: (d) cross-sectional SEM image, top-view SEM images in (e) low and (f) high magnifications, and (g-i) EDS mapping of the whole area of (f). Scale bar: (a) and (b) 1 µm, (c) 2 µm, (d) and (e) 20 µm, (f-i) 2 µm.](image-url)
Fig. S4 (a)). However, if the SiO$_2$ layer is too thick (>1 µm), the MAI vapor cannot efficiently penetrate through it so that no NWs can be obtained on the surface. It is noted that the crystals on the surface in Supplementary Fig. S4 (c) are resulted from the direct deposition of MAI on the surface. Meanwhile, when SiO$_2$ is deposited under much higher rate, such as 50 Å/s, the SiO$_2$ layer becomes much denser so that less NWs coverage rate on the surface is observed (Supplementary Fig. S4 (f)). The effect of PAM pore size on the perovskite NWFs is also investigated, as shown in Supplementary Fig. S5. It is found that smaller pore diameter PAMs result in thinner perovskite NWs, as well as lower NWs coverage rate. The reasons can be attributed to the much denser SiO$_2$ film on top of such PAMs, and less Pb seed amount within PAM channels.

This MSG method is also generic to grow different perovskite materials. As shown in Fig. 3 (a), two different sets of perovskite NWFs have been obtained, namely MAPbI$_3$ (left) and MAPbBr$_3$ (right). The SEM image of the obtained MAPbBr$_3$ NWFs is shown in Supplementary Fig. S6, exhibiting the similar morphology with that of the MAPbI$_3$ NWFs. Compared with the well-known brownish MAPbI$_3$ thin films, the MAPbI$_3$ NWFs looks more blackish because of the enhanced light absorption/trapping in the nanostructure, which is beneficial for PDs and solar cells [50–54]. The XRD patterns of the grown MAPbI$_3$ and MAPbBr$_3$ NWFs in Fig. 3 (b) illustrate the cubic phase nature of those NWs, matching with the NWs morphology revealed from the SEM images. Light absorption and photoluminescence (PL) spectra are shown in Fig. 3 (c) and (d), respectively. Both the absorption edges and PL peak positions are consistent with the reported values for MAPbI$_3$ and MAPbBr$_3$ NWs and thin films [55]. Meanwhile, the narrow PL spectra, with full-width at half-maximum (FWHM) values of 33 nm and 25 nm for MAPbI$_3$ and MAPbBr$_3$ NWFs, reveal their high crystal quality as well. This growth method also has high potential to grow other NWs when a self-catalytic VSSR is applicable, such as indium-catalyzed growth of InP NWs [56].

To reveal the light absorption behaviors, 3D perovskite NWFs are simplified to four NWs network and Finite-Difference Time-Domain (FDTD) simulation is carried out, as shown in Supplementary Fig. S7 (a). Specifically, wavelength-dependent absorption spectrum and cross-sectional $E^2$ distribution are simulated and shown in supplementary Fig. S7. It is found that no obvious antenna enhancement effect is observed, which is different from reported nanostructures [57–59]. It can be attributed to the non-uniform NWs morphology and disordered NWs distribution.

Fig. 4 (a-c) show the TEM images of a Parylene C-coated MAPbI$_3$ NW under different magnifications, where it can be seen that a 50 nm thick Parylene C layer is uniformly coated on a 160 nm thick MAPbI$_3$ NW. It can be also confirmed by the EDS line scan result shown in Supplementary Fig. S8. Meanwhile, the NW maintains its good crystallinity after Parylene C coating as revealed by the good lattice in the high-resolution TEM image (Fig. 4 (c)) and the Fast Fourier transform (FFT) result in the inset. Fig. 4 (d) are EDS mapping results of the whole area in Fig. 4 (b), which confirms the uniform Parylene C@MAPbI$_3$ composition distribution and well-engineered morphology.

Ag NWs have been demonstrated with good conductivity even under certain strain [60]. Hence, to fabricate stretchable PD device, Ag NWs are drop-coated onto the perovskite NWFs/PDMS chip with a well-designed Kapton tape as shadow mask to pattern the Ag NWs electrode. The effective device area is around 0.7 × 0.7 mm$^2$, which can be confirmed by a top-view SEM image of the device shown in Fig. 5(a). Zoomed-in SEM images in Fig. 5 (b) and (c) show the perovskite NWs embedded in PDMS and the top AgNWs electrodes, respectively. Subsequently, the PD performance without strain is firstly characterized. Fig. 5 (d) shows the current – voltage (I-V) curves under different excitation white light intensities (P). The maximum $I_{\text{light}}/I_{\text{dark}}$ can reach $1.1 \times 10^5$ under ~2 V bias and 5.5 mW cm$^{-2}$ conditions. The linear curve reveals the Ohmic contact between MAPbI$_3$ and the AgNWs.
electrode. Meanwhile, the carrier transportation within the device relies on a physical contact between neighboring MAPbI$_3$ NWs, contributing to an ultra-low dark current value (8.57 nA cm$^{-2}$ at −15 V bias, 0.18 nA cm$^{-2}$ at −2 V bias). It enables the device with an excellent sensitivity to weak light with a light intensity of 0.84 μW cm$^{-2}$, which is limited by the measurement setup. The sensitivity already exceeds that of a commercially available silicon photodiode. Fig. 5(e) and Supplementary Fig. S9 show the dependence of the photocurrent ($I_{\text{photo}} = I_{\text{light}} - I_{\text{dark}}$) and responsivity ($R$) on the illumination light intensity under −15 V and −2 V bias, respectively, indicating a perfect linear power-law relationship ($I \sim P^{0.71}$). The maximum device responsivity can reach 455.3 mA W$^{-1}$ and 70.0 mA W$^{-1}$ under −15 V and −2 V bias, respectively. In addition, the specific detectivity ($D^*$) of the device is calculated by using the following formula [20]:

$$D^* = \frac{R}{\sqrt{2eJ_{\text{dark}}}}$$  \hspace{1cm} (1)

where $J_{\text{dark}}$ is the dark current density. It is found that, under −15 V and −2 V bias, the device demonstrates 8.69 × 10$^{12}$ and 9.22 × 10$^{12}$ Jones specific detectivity, respectively. To the best of our knowledge, the device performance, especially the dark current level, ON-OFF ratio, and the specific detectivity, have already surpassed that of all perovskite-based and most of other materials-based stretchable PDs (Table S1). Fig. 5(f) shows the time-domain current values with periodically ON and OFF light illumination, where the device response speed can be extracted from the zoomed-in curve (Fig. 5(g)). Under 5 V bias and 15.75 μW cm$^{-2}$ illumination, a rise and fall time of 74 ms and 70 ms are calculated, exhibiting a decent response speed. The device also demonstrates excellent stability because of the mechanical protection from the coated Parylene C and PDMS matrix. As shown in Fig. 5(h), both the light and dark current have no obvious degradation after 250 days in the ambient environments (~ 25 °C, 40 ~ 60% relative humidity), which is significantly promising for practical applications.

To characterize the stretchable PD performance, a variety of strains are applied to the device for the measurement, as shown in Fig. 6(a). Fig. 6(b) shows the $I$-$V$ curves of a stretched device under dark and light (15.75 μW cm$^{-2}$) conditions, where decent light response is maintained even under 80% strain. Although the light current drops slightly (13%), the $I_{\text{light}}/I_{\text{dark}}$ value maintains unchanged when increasing the strain from 0% to 80% (Fig. 6(c)). The periodic time-domain light response of the device under 80% strain is shown in Fig. 6(d), exhibiting stable and consistent light response behavior similar to that of 0% strain. The excellent device performance makes them promising for practical applications in stretchable and wearable optoelectronics.

3. Conclusion

In summary, a novel MSG method has been successfully developed to grow 3D vertical perovskite NWs. The Pb metal, SiO$_2$ @PAMs, and MAI vapor work as the “seed”, “soil”, and “nutrient” during the NWs...
Fig. 5. PD performance characterization without stain. (a-c) Top-view SEM images of (a) the device with zoomed-in SEM images showing the (b) NWs embedded in PDMS and (c) Ag NWs electrode. Images (a) and (b) are taken under a back-scattering mode. Scale bar: (a) 0.5 mm, (b) 5 µm, (c) 1 µm. (d) I-V curves under different excitation white light intensities. (e) Light intensity-dependent photocurrent and responsivity of the device under −15 V bias. (f) Time-domain device current value under 5 V bias with periodically ON and OFF excitation light. (g) Response speed measurement under 5 V bias and 15.75 µW cm⁻² light intensity. (h) Device stability measurement under ambient conditions with recorded light current and dark current over 250 days period. The measurement condition: 5 V bias and 15.75 µW cm⁻² excitation light.

Fig. 6. PD performance characterization under strain. (a) Digital photos of the perovskite NWFs/PDMS membrane under different strain conditions. Scale bar: 1 cm. (b) I-V curves, (c) light (15.75 µW cm⁻²) and dark current values of the device with different strain conditions. (d) Time-domain device current value under 5 V bias with periodically ON and OFF excitation light and 80% strain.
growth, respectively, akin to the seeding growth of grass in nature. The grown NWFs have an average NWs diameter of ~200 nm and a forest height of ~50 µm with very high NW density. The obtained NWs have good crystallinity with a cubic phase. Meanwhile, this growth strategy is generic for different perovskite materials, such as MAPbI$_3$ and MAPbBr$_3$. After coating with a thin layer of Parylene C, the NWFs have been embedded into PDMS elastomers for stretchable PD device fabrication. Under 0% strain, the device has demonstrated excellent PD performance. The device dark current is as low as 8.57 nA cm$^{-2}$ at −15 V bias, and 0.18 nA cm$^{-2}$ at −2 V bias, contributing to an ultrahigh $I_{high}/I_{dark}$ value of $1.1 \times 10^4$ under 5.5 mW cm$^{-2}$ illumination. The device also exhibits a high responsivity of 455.3 mA W$^{-1}$ and specific detectivity of 8.69 $\times$ 10$^{12}$ Jones under −15 V bias. Moreover, under 80% strain conditions, the device has demonstrated negligible performance drop, especially in the aspect of $I_{high}/I_{dark}$ value. Overall, the unique growth method presented in this work provides a new strategy to obtain 3D perovskite nanomaterials with excellent optical properties. The development of the stretchable PD device here also paves a way for the practical applications of perovskite materials in stretchable and wearable electronics and optoelectronics.

4. Methods

4.1. Materials

The PDMS and curing agent kits were purchased from Dow Corning. All the other chemicals were purchased from Sigma-Aldrich and used as received without further purification.

4.2. Growth of perovskite NWFs

The growth process started from PAMs fabrication via a standard anodic anodization process. Firstly, after cutting into 1 cm $\times$ 2 cm size, pure Al (250 µm thick) was cleaned with acetone, isopropyl alcohol and deionized (DI) water successively. The chip was then electrochemically polished in an electrolyte solution with HClO$_4$ and CH$_3$CH$_2$OH in a volume ratio of 1:3 for 2.5 min under 12.5 V and room temperature condition. Subsequently, a standard 200 V anodization and barrier thinning processes were carried out with the Al chip to get the required PAMs with pore size of ~200 nm, with details reported by us before [25,38,42]. It is noted that the PAM height growth rate was around 2 µm per hour. Before the VSSR growth process, electrochemical deposition (ED) of Pb precursor in PAMs channels, PAMs surface cleaning, and SiO$_2$ layer E-beam evaporation on the PAMs surface were consecutively conducted. Specifically, the Pb ED process was carried out for 1 h in the ED solution which was prepared by dissolving 13.905 g PbCl$_2$ powder in 500 mL water-DMso (1:1 in vol) mixed solvent. A three-electrode system with an alternating current (50 Hz, 9 V amplitude) from a potentiostat (SG 300, Gamry Instruments) was utilized to conduct the Pb ED. For the PAMs surface cleaning, an ion milling process was utilized to remove the overgrown Pb metals on the PAMs surface. Specifically, argon ions were accelerated by 500 V voltage in a vacuum of $1.4 \times 10^{-4}$ torr to bombard the sample surface for 30 min. The angle between the ion acceleration path and the normal of the chip was 80° to stimulate the VSSR reaction for 12 h. After that, the chip was naturally cooled down to the room temperature in Ar gas before taking it out.

4.3. Stretchable PD device fabrication

A thin layer of Parylene C (Galentis) was coated onto the perovskite NWFs to promote the PDMS curing, which was achieved with the SCS Labcote 2 (PDS 2010) vacuum deposition system. The chip was placed on a rotating platform during the process to ensure the uniform coating, where 0.15 g of dimer was loaded into the system for ~50 nm Parylene C coating. After that, the PDMS prepolymer/curing agent mixture (weight ratio = 10:1) was dropped onto the NWFs chip and annealed at 80 °C for 30 min to cure the PDMS. After cooling down to the room temperature, the PDMS was slowly peeled off from the chip, with perovskite NWFs fully embedded inside. Finally, Ag NWs were drop-coated onto the NWs@PDMS surface to work as the stretchable electrode with a Kapton tape as the shadow mask.

4.4. Characterization

SEM images were characterized using a field-emission scanning electron microscopy (JEOL JSM-7100 F) which was equipped with a Si (Li) detector and PGT 4000 T analyzer for EDS mapping of perovskite NWFs. Using an accelerating voltage of 10 kV, the elemental mappings were recorded by counting for 100 s. TEM images and corresponding EDS mapping were obtained by a double spherical aberration corrected STEM (FEI Temis Cubed G2) with 300 kV acceleration voltage and 100 pA current. Convergence semi-angle was 25 mrad, and camera length was 115 mm. UV–vis absorption spectrum was measured using a Varian Cary 500 spectrometer (Varian, USA). PL spectra were obtained with Edinburgh FS5 fluorescence spectrometer equipped with a xenon lamp. The photodetector device was characterized using a HP 4156 A Analyzer along with probe station (Sigatone, US).

CRediT authorship contribution statement


Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Data will be made available on request.

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