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Journal

Advanced Materials, 38(38)

ISSN

0935-9648

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Publication Date

2026-07-01

DOI

10.1002/adma.202521800

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RESEARCH ARTICLE

Reversible, Photo-Induced Lattice Distortions in Halide Perovskites

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Received: 3 November 2025 | **Revised:** 21 February 2026 | **Accepted:** 24 February 2026

Keywords: halide perovskite | photoinduced lattice distortion | single crystal | X-ray diffraction

ABSTRACT

The unique characteristics of soft-lattice halide perovskites (HPs) have motivated extensive research into their application in semiconductor devices. Mechanically and chemically modulated strain in monocrystalline HPs improves stability and phase purity. However, a comprehensive understanding of the photoexcitation-driven lattice distortions arising from the strong electron-phonon coupling paired with the dynamics of the A-site cation remains underdeveloped. Here, we present the transient lattice distortions occurring in single crystal MAPbBr₃, FAPbBr₃, and CsPbBr₃ in response to above-bandgap light excitation. Using an X-ray probe, we uncover reversible and hysteresis-free photoinduced lattice distortion. We find that the photoinduced distortion in the HPs is a function of the pump power, with CsPbBr₃ showing the highest resilience against lattice deformation with a 0.062% change in its out-of-plane lattice parameter. Conversely, the organic HPs unveil a stronger interaction with photocarriers resulting in more significant yet elastic distortion, with MAPbBr₃ exhibiting up to 0.3% change. We demonstrate the modality of this distortion by varying the excitation power over 20 distinct states and cycles, highlighting the suitability of HPs as building blocks for electrostriction devices. Our findings represent a key step toward establishing HPs as ideal platforms for optical and strain-driven switchable photonic devices.

1 | Introduction

The demand for faster, cheaper, and energy-efficient electronic devices has rapidly advanced technological discoveries over the last few decades. Particularly within the realm of unconventional semiconductors, halide perovskites (HPs) have garnered huge success as platforms for photovoltaics [1, 2], light-emitting diodes [3–5], and detectors [6, 7]. HPs are most commonly fabricated via solution processing [8–11], forming polycrystalline thin films on transparent conductive oxide substrates. The thermal expansion mismatch between the film and substrate as well as the polycrystalline nature of these thin films introduces a high density of defects that can limit charge extraction and promote

faster degradation routes [12–14]. Single-crystal HPs overcome these challenges through oriented growth [15, 16], absence of grain boundaries and can be free-standing, eliminating concerns arising from interfacial defects [17–20].

The soft-lattice nature of HPs facilitates unique structure-property relations such as polaron-formation [21–23], a negative thermo-optic coefficient [24], an anomalous temperature-bandgap relationship [25, 26], and an antithetical optical response upon exposure to above-band gap illumination and heat [27] distinct from conventional semiconducting materials. At the core of these properties is the pronounced electron-phonon coupling [28–30] and the highly disordered equilibrium state

of HPs [31–34]. Early macroscopic studies of polycrystalline thin films demonstrated that photogenerated carriers can drive phase segregation of the perovskite and PbX_2 phases as well as bromide and iodide-rich phases in mixed halide compositions [35–37], in addition to triggering ion migration [38–40]. The phase segregation can be partially or fully reversible once light exposure is removed, and charge carriers recombine (composition and environmental conditions dependent). More recently, nanoscale investigations have revealed that interactions of photocarriers with the anharmonic lattice induce changes in crystal symmetry enabling structurally strained and electronically polar states. For instance, transient absorption measurements have shown that optically excited phonons in 2D HPs drive the lattice to a higher symmetry phase [41], while femtosecond-resolved electron diffraction revealed that coupling of phonons to the interband excitation of nanocrystalline FAPbBr_3 reorganizes the lattice toward a more ordered structure [42]. Contrastingly, in bulk, 3D HPs, optically generated strain leading to lower symmetry states has been attributed to coupling of photocarriers with optically excited soft transverse acoustic phonon modes [43, 44]. The lower symmetry is determined by the off-centering of the A-site cation accompanied by tilting of BX_6 (B = metal, X = halide) octahedra. The octahedral bonding network largely shapes the electronic band structure of HPs, and distortions of the structural ordering have direct implications for macroscopic properties such as absorption, polarizability, and charge transport which are critical for device operation. Although prior reports have delved into the nanoscale mechanisms responsible for photoinduced lattice distortions, macroscopic demonstrations of optically induced lattice distortion in bulk HPs have been limited. Reversible and transient optical modulation of the lattice is required to establish HPs as a platform for photostriction devices.

The choice of the A-site cation has been shown to be integral to this strong lattice anharmonicity [45–47]. Perturbations to this inherently disordered system by an external stimulus such as light, heat, or electrical bias is required for use in optoelectronic applications [48–52]. However, limiting the degradation of the perovskite phase under such external stimuli has been the key challenge inhibiting the commercialization of HP-based devices [53–55]. Engineering the A-site cation is one of the many solutions proposed to stabilize the equilibrium perovskite crystal structure [56, 57]. While optimization of the elemental composition of the A-site can deliver desirable macroscopic properties, the links between the structural origins of the perturbed, out-of-equilibrium states and their dependence on the A-site cation remain elusive. Moreover, like oxide perovskites, HPs also exhibit piezoelectricity [49, 58], though at room temperature, it mainly manifests as ferroelasticity [59–61]. Mechanically and chemically engineered strain in polycrystalline HPs can improve light absorption and emission properties via band gap normalization and enhance carrier mobility and lifetime by lowering the energy barrier and defect density. Contrastingly, mechanisms governing electrostriction in single-crystal HPs and their interplay with the dynamics of the A-site cation are yet to be explored.

Here we report an unprecedented photoinduced, fully reversible, and hysteresis-free lattice distortion in the single-crystal lattice of HPs with three different A-site cations: MAPbBr_3 , FAPbBr_3 ,

and CsPbBr_3 . Using above-bandgap light as a pump and X-ray diffraction (XRD) as a probe, we systematically quantify, in situ, the distortions that occur within the crystal lattice. The observed distortions originate from interactions of the generated photocarriers with the anharmonic lattice governed by the dynamic nature of the A-site cations. Specifically, we reveal the role of the A-site cation in determining the crystal's responsivity and stability under irradiation, i.e., out of equilibrium. We uncover that HPs with organic cations, MA^+ and FA^+ , undergo a total change of 0.3% and 0.18% in the interplanar spacing, respectively, when submitted to the light excitation stimulus, a direct consequence of the weak hydrogen bonding and its interaction with the Br^- ions. Whereas the inorganic counterpart (Cs^+) exhibits the highest structural stability with 0.062% change in its out-of-plane lattice parameter due to its intrinsically higher ionic character which suppresses the degree of electron-phonon coupling. In all cases, the lattice distortion is fully reversible, indicating that the generated photocarriers induce elastic distortions in the lattice that vanish upon their recombination. We demonstrate the hysteresis-free cyclability of this transient distortion with a non-linear response to the pump power over 40 cycles. The multi-state phenomenon discovered and reported here is primarily dependent on the power density of the excitation source. These findings are critical for the future development of devices reliant on dynamic and cyclic strain-driven response. Furthermore, our proof-of-concept in situ experiments illustrate the potential of XRD-probe-based measurements as a tool to explore correlations between chemical composition and the out-of-equilibrium structural behavior of ionic semiconductor materials.

2 | Results and Discussion

We utilize in situ XRD, using above-bandgap laser excitation (532 nm) as a pump, to elucidate the roles of the organic, MA^+ and FA^+ , and inorganic Cs^+ , A-site cations on the soft-lattice structures of HP under photoexcitation (out-of-equilibrium state). The MAPbBr_3 and FAPbBr_3 single crystals were synthesized using inverse-temperature crystallization, and CsPbBr_3 single crystals were grown using the Bridgman growth method (see Methods). We chose Bridgman-grown CsPbBr_3 crystals because this growth method ensures high-quality, free-standing single crystal growth with defect densities ranging from 10^9 to 10^{12} cm^{-3} [62]. Inverse temperature crystallization is a technique commonly used to grow organic single-crystal HPs with comparable defect densities [63, 64]. Although, different growth methods are used, we find that the optoelectronic quality in terms of absorption and emission as shown in Figure S2, and the structural phase purity, as seen from all our XRD measurements, of all three crystals is comparable. Figure 1 shows the structural evolution of a) MAPbBr_3 , b) FAPbBr_3 , and c) CsPbBr_3 single crystals as the power of the pump laser is incrementally increased. Under no photoexcitation (0 mW, black curves), the Bragg peaks for all three compositions indicate high crystallinity and phase purity. The excitation power is increased in increments of 2 mW from 0 up to 40 mW. For both MAPbBr_3 and FAPbBr_3 , we first observe a shift in the peak position to smaller 2θ with a decrease in the relative intensity of the peak as the laser power is increased. The peak intensities are relative to the scan acquired under no photoexcitation. The substantial decrease in intensity indicates that there is a significant distortion under increased pump power.

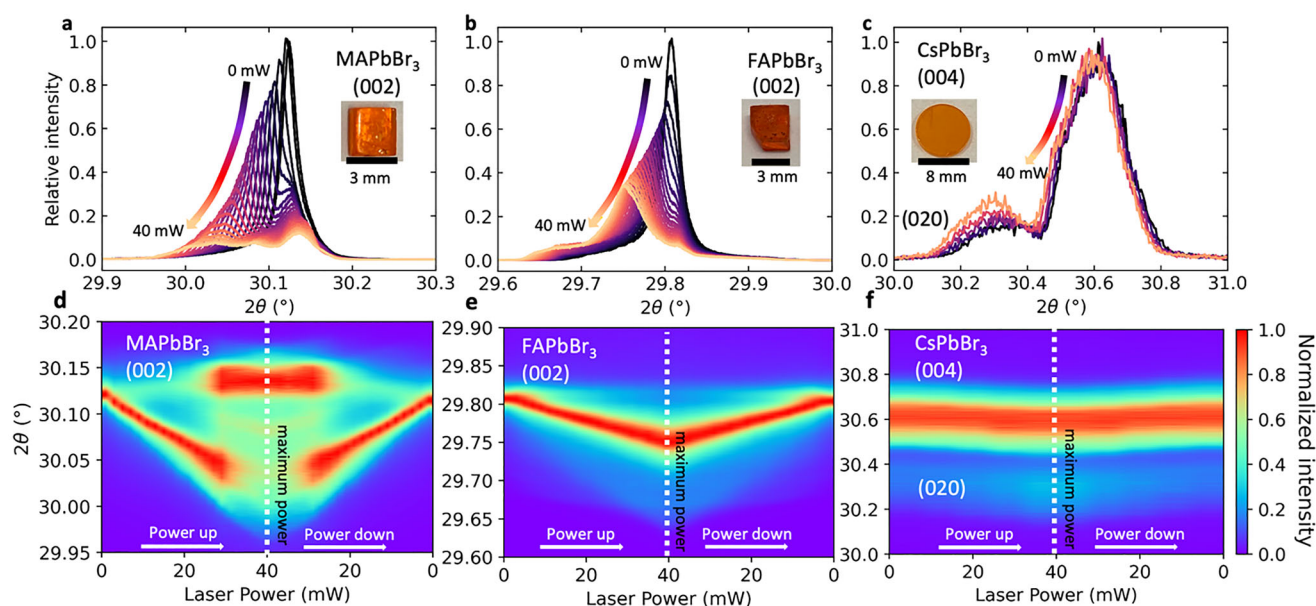


FIGURE 1 | Hysteresis-free, reversible photo-induced structural changes in single-crystal halide perovskites: In situ XRD scans with laser excitation for a) MAPbBr₃, b) FAPbBr₃, and c) CsPbBr₃. The (002) Bragg peaks are probed for MAPbBr₃ and FAPbBr₃, and the (004) for CsPbBr₃. The intensities of the Bragg peaks are relative to the scan without illumination (0 mW), where the crystal is in its equilibrium state. The laser power is incrementally increased from 0 to 40 mW in steps of 2 mW for MAPbBr₃ and FAPbBr₃, and in steps of 10 mW for CsPbBr₃. The insets are pictures of the single crystals. Heat map of the in situ XRD scans as a function of laser power with the Bragg peak intensities normalized to highlight the changes in 2θ for d) MAPbBr₃, e) FAPbBr₃, and f) CsPbBr₃. Laser excitation source: 532 nm, beam diameter = 1 mm.

At 40 mW, three distinct peaks for MAPbBr₃ can be identified. It is important to note here that X-rays penetrate deeper into the sample ($\sim 10\ \mu\text{m}$) than the pump laser ($\sim 300\ \text{nm}$) meaning that the out-of-plane XRD measurements will show some signature of the equilibrium, bulk crystal that does not interact with the photocarriers. Although out of the scope of this work, this challenge could be resolved by limiting the crystal thickness to the penetration depth of the laser. For both MAPbBr₃ and FAPbBr₃, once there is a sufficient shift in the position of the primary peak, a smaller peak at the original position is observed which we attribute to signal from the unchanged volume of the sample. The third peak for MAPbBr₃ is at a larger 2θ than the equilibrium peak signifying the large distribution of interplanar spacings identified in the distorted structure.

For FAPbBr₃, Figure 1b, the shifted primary peak holds the highest intensity at all pump powers indicating a higher degree of crystallinity compared to MAPbBr₃. The relative intensity of the shoulder peak observed at 29.7° increases with pump power, again suggesting that there is a spread in the interplanar spacings resulting in multiple distinct peaks. For CsPbBr₃, there is a minor but distinguishable shift in the 2θ positions of the (004) and (020) peaks. The intensities of these peaks remain relatively constant highlighting the structural robustness of the crystal against photoexcitation. We hypothesize that the increase in the intensity of the (020) peak could indicate a preferred strain state under photoexcitation as this crystal plane is a structural twin of the (004) plane [65]. Overall, HPs with organic cations undergo significantly larger structural distortion compared to CsPbBr₃. Between the two organic cations investigated, MAPbBr₃ shows a 90% decrease in relative intensity of the main peak as well as a larger spread in the peak width, with three distinct peaks appar-

ent upon material excitation. In comparison, FAPbBr₃ exhibits a 60% decrease in intensity of the main peak. The larger degree of lattice distortion observed for the MA-based HPs largely stems from the higher dynamic disorder of the MA⁺ cation compared to the FA⁺ cation [66, 67]. Dynamic disorder in this context is described by the rotation and reorientation of the organic cations within the inorganic octahedral cage. As a non-polar cation, Cs⁺ lacks this dynamic disorder. Previous reports have shown that the MA⁺ dipoles rotate freely, while the FA⁺ dipoles are more static, with dipole moments of 2.3 and 0.2 D, respectively [68]. Additionally, FAPbBr₃ has a more disordered arrangement of the FA⁺ ion compared to the MA⁺ counterpart which has a more ordered antiferroelectric arrangement rendering distinct photo-induced responses. Further, it has been hypothesized that the hydrogen bonding between the organic cation and halide ion is weakened by photocarriers and the corresponding reduction in density of states and Coulomb interaction at the halide site leads to shifts in the atomic positions along the Pb-X-Pb bond [69]. This priorly published result corroborates with our in situ XRD measurements of the lattice expansion. The lack of dipoles in the inorganic CsPbBr₃ has a stabilizing effect on the structure in line with the limited photo-induced distortion as shown in Figure 1c.

Between each scan with photoexcitation, we perform a scan in dark conditions (i.e., material at equilibrium) to distinguish any permanent photo-induced distortions (Figure S3). For all three compositions, the crystal structures revert to their original state with a full recovery of the relative intensity of the Bragg peaks highlighting the elastic nature of the photoinduced lattice distortion. This result directly implies that the generated photocarriers are driving the observed structural changes. Once

the photoexcitation is ceased, the photocarriers recombine and the lattice's equilibrium formation is restored. Following the increase in the laser power, we then decrease the pump power back down from 40 to 0 mW with the same profile (i.e., 2 mW increment, dark scan after each illuminated scan) (Figure S4). Figure 1d–f are heatmaps of the complete pump power profile for MAPbBr₃, FAPbBr₃, and CsPbBr₃, respectively. The intensities are normalized to 1.0 to bring focus to the change in 2θ . In the case of MAPbBr₃, as noted before, there is a large spread in 2θ . The 2θ position of the primary peak monotonically shifts to smaller 2θ , and at 32 mW pump power, the third peak at slightly larger 2θ becomes the dominant one. Contrastingly for FA, we observe a broadening of the primary peak, however, the primary peak dominates across all pump powers. The crystal structure of the HPs, especially along the out-of-plane direction which reflects the lengths of the Br–Pb–Br bonds, is intricately linked to the electronic band structure. The bandgap of HPs is primarily determined by the Pb–Br bond. Hence, any changes in the bond length are expected to also shift the bandgap which can alter the absorption, emission, and exciton binding energies of the crystal. Such reversible optoelectronic changes are useful in sensing, limiting, and switching application. In the context of our study, MAPbBr₃ exhibits a larger structural change under illumination characterized by the larger spread in 2θ values as seen in Figure 1a. While FAPbBr₃ (Figure 1b) undergoes a larger lattice expansion when photo-excited, the dominance of a single peak points to an overall lower degree of distortion. This suggests a stronger photon–lattice interaction in MAPbBr₃ than FAPbBr₃. Because the underlying mechanisms for the photoinduced changes in the two organic compositions are similar, we expect similar optoelectronic responses but likely with a larger response in MAPbBr₃. It is important to note that while MAPbBr₃ and FAPbBr₃ are cubic at room temperature, CsPbBr₃ is orthorhombic and additionally lacks the dynamic disordering present with the organic cations. While the changes in the Pb–X bonding still have implications for the electronic band structure of CsPbBr₃, the optoelectronic response of CsPbBr₃ is likely to be triggered in different energetic regimes as determined by the phonon dispersion [27, 70–72, 30].

Both MAPbBr₃ and FAPbBr₃ have a nominally cubic (*Pm3m*) phase at room temperature [73], while CsPbBr₃ has an orthorhombic (*Pnma*) structure [74]. Figure 2 shows the equilibrium and photoinduced, out-of-equilibrium structures for the three HPs. We hypothesize that the photo-induced distortion arises from the interactions of the generated photocarriers with the anharmonic, soft lattice. Due to the strong electron–phonon coupling, photocarriers can deform the lattice as they propagate through it. Measurements in dark condition and with a pump laser with photon energies below the band gap (685 nm) both show no structural changes (Figure S5) highlighting that absorption of light and generated photocarriers are the main cause for the distortion. As evidenced by the in situ XRD measurements, the photo-irradiation causes a large distortion in MAPbBr₃ (Figure 2a,b). Even though there is a significant decrease in the peak intensity, we are still able to distinguish the presence of the (002) peak. A decrease in diffraction intensity can arise from two reasons: 1) loss of phase or 2) loss of crystallinity, both of which point to the lack of diffracting planes at the given 2θ angles. We perform in situ XRD measurements with a larger 2θ range of 5°–35° and verify that no additional phases are

formed in the distorted structure (Figure S6). In polycrystalline thin films, photoinduced phase segregation is a widely observed phenomenon and peaks for the PbX₂ phase are easily identified in XRD measurements [75]. We do not observe the emergence of any new peaks under photoexcitation across this larger 2θ range, ruling out phase segregation or loss of phase as a sounding explanation for reduced peak intensity. Therefore, loss of crystallinity due to the induced distortion is the more probable cause of the reduced intensity.

To further understand the observed loss in intensity, we consider the alternative rationale of diminished crystallinity. During single-crystal XRD measurements, the tilt and rotation of the sample are aligned to bring the desired out-of-plane direction perpendicular to the measurement axis. This alignment necessitates that only the chosen family of planes, i.e., {00l} in our case, will diffract in a regular $2\theta/\omega$ scan. Because the samples' alignment was kept the same for each illuminated measurement, any loss of crystallinity, in other words, any deviation from the equilibrium cubic structure, would result in a decreased intensity because the {00l} diffracting planes are no longer aligned such that they are perpendicular to measurement axis. This situation is schematically portrayed for MAPbBr₃ in Figure 2b, where the increased octahedral tilting and lattice expansion are exaggerated for clarity. The degree of octahedral tilting and plane-by-plane distortion shown cannot be directly inferred from our XRD measurements. However, we estimate this tilting based on the known dynamic of the halide perovskite family, their short-range lattice distortions observed via atomic pair distribution function and prior nano-diffraction measurements [44, 76]. FAPbBr₃ undergoes a relatively smaller degree of distortion mainly arising from a lattice expansion as shown in Figure 2c,d. The presence of 3 distinct peaks is also observed for FAPbBr₃ indicating a significant degree of octahedral tilting and local distortions beyond just the lattice expansion. Conversely, CsPbBr₃ exhibits a small change to its crystal structure with mainly a lattice expansion accompanied by reduced octahedral tilting depicted in Figure 2e,f, respectively.

To address the potential concern of lattice expansion emerging from laser-induced sample heating, we calculate the estimated rise in temperature for the conservative case where all the energy from the laser at 20 mW is absorbed in the crystals as heat (See Note S1). This assumption provides the upper limit of the temperature rise as a significant portion of the laser energy will be absorbed in generating photocarriers. Our calculations show that the top surface of the MAPbBr₃, FAPbBr₃, and CsPbBr₃ crystals will heat to 46°C, 50°C, and 45°C, respectively. As the thermal properties of the three compositions are comparable, we expect to see a similar response to any laser-induced heating [77, 78]. Next, we perform controlled, temperature-dependent XRD measurements on the single crystals from 25°C to 50°C (Figure S7) and observe the expected shift in 2θ to lower values indicative of thermal expansion. At 50°C, there is shift of up to 0.035°, 0.065°, and 0.04° in the peak positions of MAPbBr₃, FAPbBr₃ and CsPbBr₃, respectively. However, under illumination at 20 mW, we report maximum peak shifts of 0.06°, 0.09°, and 0.02° for MAPbBr₃, FAPbBr₃, and CsPbBr₃, respectively. The maximum peak shifts are determined as the difference between the positions of Peak 1 under no illumination and Peak 2 for FAPbBr₃ and Peak 1 for both MAPbBr₃ and CsPbBr₃ at 20 mW.

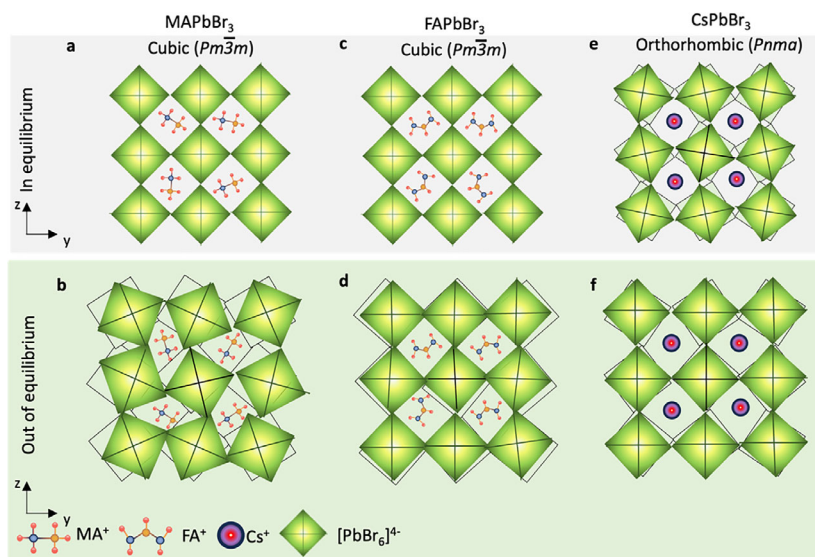


FIGURE 2 | Photo-induced lattice distortions for single-crystal halide perovskites with three different cations (MA^+ , FA^+ , and Cs^+): Schematic showing the (top) equilibrium and (bottom) out-of-equilibrium distorted crystal structures for a,b) MAPbBr_3 , c,d) FAPbBr_3 , and e,f) CsPbBr_3 , respectively. Out of scale for clarity.

For the organic cations, the peak shifts under illumination are almost twice as much as heating at 50°C , therefore, we expect a negligible contribution of laser heating in our illuminated experiments. Additionally, on heating there is a negligible change in the intensities of the Bragg peaks indicating a uniform, isotropic lattice expansion that preserves the alignment of the diffracting planes. Whereas, on illumination, there is a significant loss of intensity and change in shape of the diffraction peaks. This observation corroborates with our attribution of loss of crystallinity to lattice interactions with photo-generated charge carriers as opposed to simply laser heating. While there is likely some convoluted contribution from laser heating too due to poor quantum efficiencies in HPs, the two stimuli (light and heat) clearly elicit different structural responses.

We further analyze the in situ XRD scans by fitting pseudo-Voigt models to each measurement, with the complete models being comprised of up to three individual fits. Under dark conditions, one fit is sufficient for representing the data well. For MAPbBr_3 , at 10 mW, there is a large enough shift in 2θ that two distinct peaks, labelled Peak 1 and Peak 2, are fit to the data as shown in Figure 3a. Figure 3b–d shows the fits for scans taken at 20, 30, and 40 mW pump powers, respectively. At 20 mW, there is a greater separation between the two fitted peaks, and this trend progresses with a separation wide enough to distinguish a third peak, labelled as Peak 3 (Figure 3c). Peak 2 becomes dominant at 40 mW. As mentioned above, Peak 3 is likely a signature of the undistorted crystal. The larger interaction volume of the X-rays compared to the laser inevitably captures bulk information and presents the average structure over this interaction volume. Additionally, it is important to note that the relative intensities of Peaks 2 and 3 remain fairly consistent further affirming the averaging effect observed. Figure 3e displays the intensities of each of the three peaks from the pseudo-Voigt fits summarizing the trend observed across all measured pump powers. The detailed fits for all measurements are shown in Figures S8 and S9 for increasing and decreasing pump powers, respectively.

A comparison of the three cations shows that each exhibits a unique interaction with light. A similar analysis is performed for FAPbBr_3 as shown in Figure 3f–j (detailed fits of all pump powers are shown in Figures S10 and S11). We identify the inherent presence of a shoulder peak (labeled Peak 2) in the dark scan for FAPbBr_3 , which becomes more pronounced under illumination and remains constant as the pump power is increased. We attribute this shoulder peak to defects inherently present in the single crystal. We observe that these defect states, that have likely introduced additional strain to the equilibrium lattice, interact modestly with the laser induced changes due to the relative consistency of the peak intensity across the pump power profile. Peak 3, as mentioned for MAPbBr_3 , is likely representative of the undeformed lattice in the interaction volume. As shown in Figure 3j, the predominant photoinduced change in FAPbBr_3 manifests as a reduction in peak intensity and shift to smaller 2θ values. The width of the primary peak does not broaden as much as it does for MAPbBr_3 . Finally, we perform the same analysis for CsPbBr_3 (Figure 3k–o; Figure S12) and find the relative intensities and positions of the peaks largely unchanged. Figure S13a–c shows the change in peak position as a function of increasing and decreasing laser power for MAPbBr_3 , FAPbBr_3 , and CsPbBr_3 , respectively. For all three cations, we report a recovery percentage of 99% across 40 cycles between the two profiles, indicative of a largely hysteresis-free phenomenon. The measurements are performed under ambient conditions giving rise to the possibility of air-induced degradation. Figure S14 shows the peak positions (a–c) and relative intensities (d–f) of the three crystals remain consistent under no illumination. The dark scans are taken after each illuminated scan to ensure that the crystals revert to their equilibrium structure. Hence, we confirm that there is no material degradation. The reversible lattice distortions dominate over any degradation owing to the higher-quality, phase-purity, monocrystalline nature, and lack of interfacial concerns of the bulk, free-standing crystals, compared to solution-processed polycrystalline thin films. Overall, CsPbBr_3 shows superior stability to photoinduced distortion. However, the

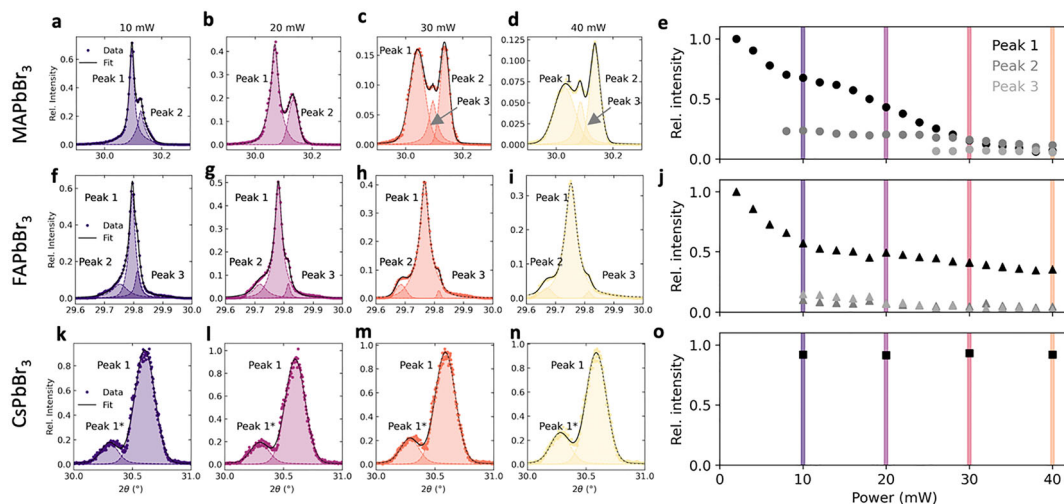


FIGURE 3 | Evolution of lattice distortion in halide perovskites as a function of laser power: Measured XRD data for the (002) Bragg peak (scatter plot) is fit with pseudo-Voigt fits (solid lines) comprising of up to 3 peaks for laser illuminated scans at laser powers of a) 10, b) 20, c) 30, and d) 40 mW for MAPbBr₃. e) Relative intensities of the three peaks comprising each pseudo-Voigt fit as a function of laser power. Same analysis for f–j) (002) Bragg peak for FAPbBr₃ and, k–o) for (004) Bragg peak for CsPbBr₃. Peak 1* refers to the (020) plane for CsPbBr₃, not included in this analysis.

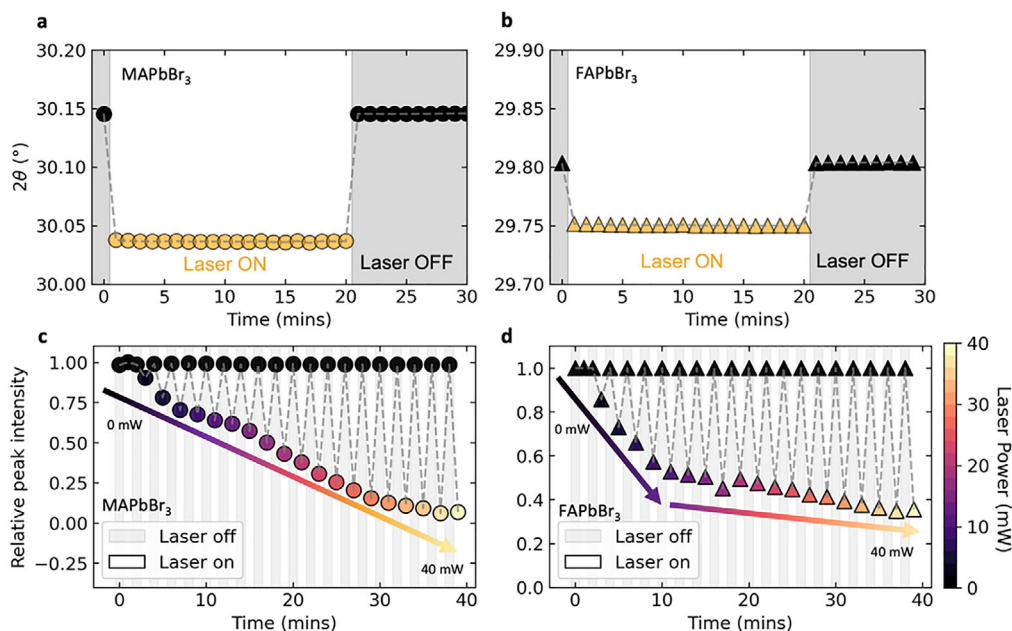


FIGURE 4 | Stability, cyclability, and multi-state switchability. Measured change in 2θ ((002) Bragg peak) with (yellow symbols) and without (black symbols) laser excitation for a) MAPbBr₃ and b) FAPbBr₃. Measured change in (002) Bragg peak intensity (relative to the first laser OFF measurement) under cyclic (OFF/ON) laser excitation for c) MAPbBr₃ and d) FAPbBr₃ while varying laser power.

distinction in the structural responses of the three organic cations underscores the varying effects of their coupling to the lattice, and the crucial role of the A-site cation in controlling the lattice dynamics.

An important metric for switchable devices is their responsivity and cyclability [27]. To this end, we investigate the temporal behavior of the photoinduced distortions in FAPbBr₃ and MAPbBr₃. Figure 4a,b shows changes in 2θ as a function of time for MAPbBr₃ and FAPbBr₃, respectively. Here, each XRD scan took 1 min to complete at the detection capabilities of the

tool. The 2θ positions of the primary peaks (Peak 1) for both crystals are shown. After an initial dark scan (black symbols), the laser power was set to 40 mW, and 20 repeated scans were acquired. Following the 20 illuminated scans, 10 scans with no photoexcitation were taken. The corresponding $2\theta/\omega$ scans are shown in Figure S15. Over the minutes-long time scale probed here, the crystals exhibit no further changes to their crystal structure in both the equilibrium and out-of-equilibrium states. This consistency emphasizes the stability of the crystals in both states and further negates any concerns surrounding laser heating. Due to the low thermal conductivity of HPs, any heating

from constant exposure to the laser would be noticeable over this time scale. Given the limited photoinduced change observed in CsPbBr_3 , we expect a similar structural resilience in both states for CsPbBr_3 crystals too. Overall, we demonstrate that the stability of the out-of-equilibrium states of the HP single crystals emulates that of the equilibrium configuration, for all three cations, which is crucial for enabling robust multi-state operation in future electrostriction devices.

Finally, we cycle the crystals between the equilibrium and the photoinduced out-of-equilibrium states as shown in Figure 4c,d for MAPbBr_3 and FAPbBr_3 , respectively. We simultaneously demonstrate both switchability and power-dependent modality in MAPbBr_3 and FAPbBr_3 by cycling the pump laser on and off. With each laser on scan, the laser power is increased to track the changes in lattice parameter and peak intensity. The power of the 532 nm laser is increased by 2 mW with each cycle resulting in a larger change in the intensity. The peak intensity reverts to its original value for each dark scan indicating a completely recovery under dark. For MAPbBr_3 , both peak intensity and peak position follow a decreasing trend with increasing power, with a unique intensity and position corresponding to each laser power. FAPbBr_3 , instead, shows a sharper decrease in the intensity at laser powers up to 10 mW followed by a plateau in intensity. Therefore, MAPbBr_3 proves more suitable for use in future devices requiring finer tuning of the accessible operating states. Most importantly, the complete recovery to the equilibrium state upon each laser off scan is a testament to the reliable cyclability exhibited by all the HP single crystals investigated in this work.

3 | Conclusions

In summary, we experimentally demonstrated fully reversible and hysteresis-free photoinduced lattice distortions in single-crystal HPs. We resolved the impact of the A-site cation in determining the nature and extent of the photoinduced distortions. MAPbBr_3 shows the highest degree of deformation, expressed by the suppression and widening of the diffracting out-of-plane Bragg peak intensity, compared to FAPbBr_3 and CsPbBr_3 , which mainly exhibit a photoinduced shift in the peak positions. For all three cations, the equilibrium structure was retained by allowing the crystal to fully relax in dark conditions (back to equilibrium state). Even under constant illumination, there is no significant permanent degradation of the crystals. Strikingly, across the 40 cycles of incrementally increasing and decreasing pump power profiles, we report a 99% recovery of the lattice distortion highlighting the hysteresis-free nature of this photoinduced response. We showed that the physiochemical interaction of the A-site cation with the lattice is critical to the structure-property-stability relationship. MAPbBr_3 , with its higher degree of distortion under illumination, enables multiple, unique distorted states directly dependent on the pump power. Although CsPbBr_3 has the highest structural stability, the structural modulation possible in MAPbBr_3 and FAPbBr_3 make them ideal candidates for use in strain-engineered optical devices. Photoinduced optoelectronic and structural responses have also been observed in the iodide-based analogues. In fact, the tetragonal structure of MAPbI_3 enables ferroelectricity [79] and photostriction [69]. Recently,

ferroelastic domains have also been identified in orthorhombic CsPbI_3 [80]. Hence, we can anticipate similar photoinduced structural changes in iodide-based compositions, although the degree of distortion will likely be determined by the strength of the hydrogen bonding between the cation and halide ion as well as the phonon dispersion of the specific compositions. Overall, a comprehensive understanding of the light-induced transient response presented here is critical for the integration of halide perovskites in electrostriction-based, dynamic devices.

4 | Experimental Section/Methods

4.1 | Material Synthesis

4.1.1 | Synthesis of Polycrystalline CsPbBr_3

CsPbBr_3 was synthesized from commercially available pure CsBr (6N) and PbBr_2 (5N). For the synthesis of 20 g of the perovskite compound, the stoichiometric amounts of the raw materials were thoroughly mixed in a mortar, following a molar ratio of 1:1. This mixture was then loaded into a quartz ampoule with an inner diameter of 10 mm. All preparation steps were conducted inside a glovebox filled with argon gas. The prepared ampoule was connected to a vacuum pump and evacuated to approximately 10–3 mbar before being flame sealed. The synthesis of CsPbBr_3 was then carried out using a solid-state method (melting the materials together) in a muffle furnace. The sealed ampoule was heated to 650°C and maintained at this temperature for 10 h to ensure the material homogenization and the completion of the chemical reaction. Afterward, the furnace was allowed to cool to room temperature over the course of 10 h. As a result, a bright orange polycrystalline ingot was obtained.

4.1.2 | Bridgman Growth of CsPbBr_3 Single Crystal

After the synthesis, the ampoule with material was placed in a vertical three-zone Bridgman furnace and heated to a temperature of 650°C. The furnace was maintained at this temperature for 10–15 h to ensure complete melting and homogenization of the material. After that the ampoule was translated from the hot to the cold zone of the furnace at a speed of 0.5–1 mm/h to perform directional crystallization. Once the growth process was complete, the furnace was gradually cooled to room temperature for 20 h. The resulting CsPbBr_3 single crystal was free of cracks and transparent.

4.1.3 | Sample Preparation

The obtained CsPbBr_3 single crystal was cut into wafers with a thickness of 1 mm, perpendicular to the growth axis of the ingot, using a diamond wire saw. The samples were polished with sandpaper of varying grit sizes, specifically 4000, 8000, and 12 000. For the final polishing step alumina powder with a particle size of 1 μm was used. The resulting substrates were <20 nm in roughness. During the cutting and polishing process, WD-40 was used as a coolant and lubricant. Afterward, the

samples were washed in toluene to eliminate any remnants of the lubricant, polishing powder, and microparticles of the material.

4.1.4 | Synthesis of MAPbBr₃ and FAPbBr₃

For MAPbBr₃, a 1.5 M DMF solution containing equimolar PbBr₂ and MABr was prepared. For FAPbBr₃, a 1.4 M solution of equimolar PbBr₂ and FABr was prepared in a 1:1 (v/v) GBL/DMF mixture. Both solutions were stirred overnight at room temperature and then filtered through 0.22-μm PTFE filters. The filtrates were transferred to vials, placed undisturbed in an oil bath, and heated from room temperature at 5°C h⁻¹ until crystals of the desired size formed. All procedures were carried out under ambient conditions.

4.2 | Material Characterization

4.2.1 | XRD

Single crystal X-ray diffraction (XRD) was performed using a Rigaku SmartLab Diffractometer with Cu-Kα radiation. A Ge monochromator was used. The scans were collected with a step size of 0.008° at a scan rate of 0.48° min⁻¹. 0.5 mm incident and Soller slits were used to limit the X-ray beam spot on the sample. Measured data (scatter plots) were fit using a pseudo-Voigt model (solid lines). A 532 nm laser diode (Vortran Laser Technologies) with a 1 mm beam diameter was used for the laser-illuminated scans. The power of the laser was controlled using the Stradus GUI. Illuminated scans were performed at normal incidence to the out-of-plane orientation of the crystals, at ambient conditions (22°C, ~50% relative humidity). The temperature of the samples was controlled using the Anton Parr DCS 500 Doomed Heating/Cooling stage. A graphite dome was used to limit the sample's interaction with ambient surroundings. All measurements were acquired at ambient pressure.

Author Contributions

M.S.L. conceived the project. M.D. performed the XRD measurements and data analysis. B.T. and A.K. fabricated the single-crystal perovskites, while supervised by M.K. M.D. wrote the first version of the manuscript and all authors contributed to the discussion and manuscript revision.

Acknowledgements

M.S.L. acknowledges the financial support from DARPA (award # HR00112190008) and NSF (award #2415023). M.D. thanks the 2024 and 2025 UC Davis Summer Graduate Student Researcher Awards. This research used a Rigaku SmartLab thin film X-ray diffractometer in the UC Davis Advanced Materials Characterization and Testing (AMCaT) Laboratory, acquired with funding from the National Science Foundation Major Research Instrumentation (MRI) Program under award number MRI-2216198. M.D. thanks P. Escobar for assistance with the heat transfer calculations. The samples were synthesized at The SynMatLab (Laboratory for Multiscale Materials Synthesis and Hands-on Education, an ETH+ project).

Funding

DARPA (award # HR00112190008), UC Davis Summer Graduate Researcher Awards 2024, 2025.

Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Supporting Information

Additional supporting information can be found online in the Supporting Information section.

Supporting File: adma72703-sup-0001-SuppMat.pdf