Observation of Two Thresholds Leading to Polariton Condensation in 2D Hybrid Perovskites

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2D perovskites are promising materials for photonic applications, given their outstanding nonlinear optical properties, ease of fabrication, and versatility. In particular, exploiting their high oscillator strength, the crystalline form of 2D perovskites can be used as excitonic medium in optical microcavities, allowing for the study of their optical properties in the strong light–matter coupling regime. While polariton condensation is observed in different materials at room temperature, for the first time two distinct threshold processes in a 2D perovskite are observed, a material that has never shown spontaneous phase transition up to now. In particular, lasing from the bi-exciton state is also demonstrated, which contributes to populate the lower polariton branch and, at higher excitation powers, eventually leads to the formation of a polariton condensate. The emission linewidth narrowing and a spatial coherence over $50 \times 50 \mu m^2$ area is the smoking gun of a quantum coherent state formation in the 2D hybrid perovskite. The results not only show the formation of a polariton condensate in 2D perovskites but they are also crucial for the understanding of the physical mechanisms that lead to coherent phase transition in perovskite-based polariton microcavities.

In the last decade, hybrid organic–inorganic 2D perovskites have attracted considerable interest because of their intriguing optical properties,[1–3] and better moisture stability than their bulk counterpart.[4] In particular, these materials show a natural quantum well-like structure formed by an inorganic layer of $(PbX)_2^−$ octahedra (in which X is an halogen), sandwiched between bilayers of intercalated alkylammonium cations. In this type of structure, the organic cation behaves as a potential barrier, while the excitons are confined within the inorganic layer.[5,6] The nature of inorganic and organic components defines the quantum well architecture that can be opportunely engineered by changing the chemistry of the synthetic process in order to tailor the optical bandgap and the exciton confinement.[7,8]

Recently, Chong et al.[9] have highlighted the difficulties in achieving optical gain in a bare single crystal of 2D perovskite, predicting a theoretical bi-exciton amplified spontaneous emission (ASE) threshold of $\approx 1.4 \text{ mJ} \cdot \text{cm}^{-2}$ that goes beyond the crystal damage threshold.

As we demonstrate in this paper, this limit can be overcome embedding a single crystal in an optical microcavity exploiting the strong light–matter coupling regime, that is, the formation of exciton-polaritons.[3] These bosonic quasi-particles—arising from the strong coupling between excitons and photons—have unique properties inherited from their bare components: The polariton mass is three order of magnitude lighter than the one of the exciton thanks to the photonic component,[10] whereas the polariton Kerr nonlinearities—that are up to four orders of magnitude higher than those of standard nonlinear optical media[11]—are inherited from the excitonic component. These peculiar properties make microcavity polariton an ideal candidate for integrated photonic circuits and electro-optical devices.[12–14] Furthermore, many fascinating physical phenomena related to quantum fluids, such as polariton condensation,[15,16] superfluidity,[17,18] and quantized vortices dynamics,[19] have been studied both in inorganic and organic polariton devices. In particular, 3D[20] and 2D[21–24] perovskites are widely used as active medium in optical microcavities, leading to the formation of a strong light–matter system stable at room temperature. Moreover, polariton lasing and condensation have been described by Su et al.[25,26] in 3D CsPbX₃ perovskites, although exciton-polariton condensation...
in 2D perovskite has never been observed before. As pointed out by Schlaus et al.,\cite{27} it is not trivial to unambiguously distinguish polariton condensation from lasing emission in materials with multiple exciton levels, such as in lead halide perovskites, due to the presence of competitive population/depopulation kinetics.

Here, we investigate the optical properties of the 2D single-crystal perovskite polariton microcavity at cryogenic temperature (4 K) demonstrating the presence of two different coherent states forming at two distinct power thresholds: a first lasing action from the bi-exciton state contributes to the subsequent collapse of the polariton population to a macroscopically coherent polariton condensate, characterized by a second threshold at higher pump powers. Our results bring out the complexity of kinetics in perovskite-based systems and pave the way for the study of macroscopic quantum coherent states in 2D perovskites-based polariton microcavities.

The phenethylammonium lead iodide perovskite (C₆H₅(CH₂)₂NH₃)₂PbI₄ (PEAI) single crystals are synthesized by antisolvent vapor assisted crystallization method on a glass substrate.\cite{28} Millimeter-sized flakes having a thickness varying from few to ten micrometres are mechanically exfoliated to improve the surface quality of crystals and to obtain the desired thickness (see Experimental Section for further details). The photoluminescence is recorded using a 50 fs pulsed excitation centered above the band gap (2.640 eV) in reflection configuration. In Section S1 and Figure S1, Supporting Information, the reflectance and emission spectra of a 70 nm-thick PEAI single crystal at 120 K are reported. The emission peak is centered at 2.364 eV with 50 meV full width half maximum (FWHM). In the reflectance spectrum, we observe a single peak at 2.366 eV, exhibiting a typical minimal Stokes shift of the excitonic emission.\cite{29}

At 4 K (Figure 1a), the photoluminescence and reflection spectra are more structured, showing the presence of three different peaks. In the emission spectrum at the lowest incident fluence (18 μJ/cm²), the peaks centered at E₁ = 2.341 eV and E₂ = 2.330 eV are associated to two electron–phonon replicas of the lowest, exciton state\cite{30} while the peak at E₃ = 2.300 eV is attributed to Frenkel defects.\cite{9} When increasing the pump power, a narrow peak appears in the emission spectrum at E_{biex} = 2.290 eV with FWHM ≃ 10 meV (Figure 1a, green line), which is usually attributed to the bi-exciton state,\cite{9,31,32} despite its true nature is yet under debate.\cite{33} At low pump fluence, the broad defect peak at E₁ = 2.300 eV is energetically overlapped to the bi-exciton emission, that is weaker than Frenkel defects.

![Figure 1.](image-url) a) Normalized photoluminescence spectrum of 70 nm-thick single crystal of PEAI at 4 K under 18 μJ/cm² 488 nm-cw laser excitation (red line), compared with the normalized photoluminescence signal under 1900 μJ/cm² 50 fs-pulsed laser excitation (green line). The black line is the normalized reflectance spectrum. Three different peaks are observed (E₁ = 2.300 eV, E₂ = 2.330 eV, and E₃ = 2.341 eV) in the photoluminescence spectrum with cw laser excitation. At high pump fluence (i.e., pulsed excitation), a narrow peak appears at E_{biex} = 2.290 eV, which is attributed to the bi-exciton emission. b) Comparison of the emission intensities of the two different emission signals at E_{biex} = 2.290 eV (orange and green dots) and E₃ = 2.341 eV (blue dots) as function of the pump fluence. The intensity of the bi-exciton state keeps increasing while the exciton state saturates at increasing pump power. Orange dots represent the defect emission signal at E_{biex}, which hides the bi-exciton emission signal at low pump fluence (up to ≃ 600 μJ/cm²) and saturates after ≃ 1000 μJ/cm² (data not shown in the graph). c) Sketch of the optical microcavity sample. PEAI flakes are embedded in an optical microcavity made by a bottom DBR and a top silver mirror. d) Energy dispersion of the emission of a 3 μm-thick single crystal embedded in a planar microcavity, in which multiple lower polariton branches are visible. The blue dashed line is the theoretical dispersion of the eigenstates of the coupled system resulting from the strong coupling between the excitonic transition at E_{ex} = 2.355 eV (red dashed line) and the optical modes in green dashed lines (the two dispersions correspond to the TE and TM polarized modes).
emission (Figure 1b, orange dots). At increasing pump powers (above \( \approx 600 \mu J/cm^2 \)), the bi-exciton emission becomes visible (Figure 1b, green dots) above the defect signal and its intensity continuously increases. The bi-exciton state accumulates population, while the emission of all the other states saturates, as previously reported in ref. [9]. Our measurements are performed on the bare material, outside any microcavity resonator, however the trends of the photoluminescence signals with respect to pumping power are fully consistent with the ones reported by Booker et al.\[34\] (Figure S2, Supporting Information), where the authors claim microcavity bi-exciton lasing. We also report a bi-exciton emission linewidth even narrower than the one of the lasing signal reported in ref. [34], despite, in our case, the material is definitely not in the lasing regime. Therefore, in our opinion, the signal observed in ref. [34] should be interpreted as incoherent emission filtered by the optical microcavity rather than a lasing signal (see Section S2, Supporting Information, for further details).

We further investigate PEAI perovskite embedded in an optical microcavity. In this case, single crystals are grown on a Distributed Bragg Reflector (DBR, Figure S3, Supporting Information) and a 80 nm-thick silver layer is evaporated on top of the perovskite crystal (Figure 1c, Supporting Information). The microcavity quality factor is \( Q > 1000 \) (Section S4 and Figure S4, Supporting Information). The photoluminescence energy dispersion (Figure 1d) shows the lower polariton branches of a 3 \( \mu m \)-thick single crystal of PEAI. Multiple lower polariton branches are due to the strong coupling (Figure S5, Supporting Information) of the lowest-energy excitons (\( E_{ex} = 2.355 \text{ eV} \)) with different optical modes, according to the microcavity free spectral range.\[21\] In Figure 1d, the blue dashed lines are the theoretical lower polariton branches resulting from the strong coupling between the exciton transition (red dashed line) and the transverse electric (TE) and transverse magnetic (TM) polarized cavity modes (green dashed lines). The Rabi splitting is \( \Omega \approx 110 \text{ meV} \) (see Section S5, Supporting Information, for detailed analysis).

A very useful insight on these light–matter levels and their related phenomenology can be obtained studying their nonlinear response at increasing pump fluences. All the optical measurements are performed in reflection configuration at cryogenic temperature, exciting and collecting the signal from the DBR side.

Figure 2a–c shows the power dependence of the polariton emission dispersion of the perovskite microcavity under a 50 fs pulsed laser (Gaussian spot size with FWHM=5 \( \mu m \)). At low fluence \( F = 5 \mu J/cm^2 \) (Figure 2a), the emission is intense at high in-plane wavevectors of the lower polariton branch (LPB). The FWHM of the polariton dispersion below the lasing threshold

**Figure 2.** a–c) Energy versus momentum emission intensity maps for three different incident pump fluences, exciting a 3 \( \mu m \)-thick perovskite single-crystal embedded in a planar microcavity (50-fs pulsed excitation at 2.64 eV). The red dashed lines are a guide to the eye for the TE and TM polariton modes. The black dashed line indicates the bottom energy of the lower polariton branch at very low excitation power. a) At 5 \( \mu J/cm^2 \), the emission is intense at high-energy and high in-plane wavevectors. b) Increasing the incident fluence up to 50 \( \mu J/cm^2 \), the bi-exciton state starts lasing and we observe an intense peak appearing at about \( E = 2.295 \text{ eV} \). c) Further increasing the pump fluence, the whole emission collapses to the bottom of the lower polariton branch (\( k \approx 0 \mu m^{-1} \)), generating a polariton condensate. d) Integrated intensity of the emission as function of incident pump fluences. Inset: Zoom for weak excitation intensities. Two different thresholds are clearly visible, the first one at \( F = 50 \mu J/cm^2 \) (black dashed line) indicates the bi-exciton lasing activation, and the second one at \( F = 200 \mu J/cm^2 \) (green dashed line) is associated to the formation of the polariton condensate at the lowest energy state. e) Energy blueshift of the polariton dispersion (blue dots), as a function of incident fluences reported in the left axis. Above the first threshold (black dashed line), the bi-exciton lasing emission starts to appear (red dots) at different energies along the polariton dispersion, in the right axis. At the onset of the polariton condensate (green dashed line), the population collapses to the bottom of the polariton dispersion and follows the blueshifts of the polariton mode. f) The FWHM of the bi-exciton lasing emission (red dots) increases at increasing pump power above the first threshold (at \( F = 50 \mu J/cm^2 \)) and it is broader than the FWHM of the polariton mode below the lasing threshold (blue dots, before the black dashed line)—the FWHM of the bi-exciton spontaneous emission is \( \approx 10 \text{ meV} \). For pump fluences above \( F = 200 \mu J/cm^2 \) (green dashed line), the FWHM of the polariton dispersion (blue dots after the green dashed line) narrows due to the onset of the polariton condensate. The red dashed line indicates the optical setup resolution (1.2 meV).
(blue dots before black dashed lines, Figure 2f) is ≃ 2 meV and is consistent with the value reported in Figure S4, Supporting Information—the fluctuations of the polariton linewidth are within the measurement uncertainty (± 0.3 meV).

Increasing the incident pump power, we observe a first threshold at about \( F = 50 \mu J/cm^2 \) where an intense emission peak appears at about \( E = 2.295 \) eV, a few meV above the bottom of the LPB. We attribute this peak to the onset of a lasing signal, given the narrowing of the linewidth and the nonlinear increase of the emission intensity (Figure 2d,f). Laser linewidth broadening and peak blueshift are observed at increasing pump power. This behaviour leads to the conclusion that this signal is related to the bi-exciton lasing action rather than to the polariton condensate emission since the system is strongly out of equilibrium, as confirmed by the lasing energy—which is higher than the bottom of the LPB. Moreover, as there is no thermalization (Figure S6, Supporting Information), the lasing action is given by the cavity gain at the uncoupled bi-exciton state, which emission energy is resonant to a specific microcavity mode (\( E_{\text{em}} = 2.290 \) eV). Considering the population accumulation to the bi-exciton state observed in the out-of-cavity perovskite layer (Figure 1b) at increasing pump powers, the fact that the bi-exciton state is the first one to achieve the lasing threshold (i.e., the population inversion) is also theoretically expected.\(^9\) Note that the energy and angle of emission of the bi-exciton lasing change with increasing power (Figure 2e, red dots between black and green lines), possibly due to different gain conditions at higher carrier concentration. Moreover, this lasing blueshift does not relate to a blueshift of the lower polariton dispersion, and the bi-exciton emission peak is broader than the polariton linewidth (about 2.3 meV, Figure 2f). This demonstrates that both the bi-exciton emission linewidth and the energy blueshift are independent from the polariton states. To further confirm such interpretation, we observe at higher fluences (\( F = 200 \mu J/cm^2 \)) a second phase transition to a higher coherent state at the energy minimum of the LPB associated with a dramatic increase of the emission intensity (Figure 2d). As it can clearly be observed in Figure 2c, the whole population collapses to the lower energy state, with almost no signal from higher energy states—also the bi-exciton lasing action turns off—as predicted in case of bosonic stimulation (Figure S6, Supporting Information). The population energy distribution in this new regime is very different from the one of the bi-exciton lasing (Figure S6b, Supporting Information). When the bosonic condensate is established at the bottom of the lower polariton branch, the FWHM narrows indicating the increase of the coherence time, and its spectral width goes below our setup resolution (1.2 meV, red dashed in Figure 2f). In this regime, the energy shift of the emission (Figure 2e) is consistent with a partial saturation of the coupled exciton state.\(^3\)

These results also demonstrate that the physics of polariton condensation in such complex materials with many exciton levels, cannot be solely explained by considering the physics of a two level system.

We point out that no photodegradation of the sample has been observed with pump fluence up to 300 \( \mu J/cm^2 \) and for an exposure of about 1 h, while for higher pump fluence (3000 \( \mu J/cm^2 \)), photodegradation occurs for a relatively shorter exposure time (≥ 10 min). Although the high pump fluence values that have been used during the experiments, the actual carrier density is considerably lower than the Mott critical density.\(^3\)

Furthermore, the polariton condensate forms exclusively when the energy of the bottom of the polariton mode is close to the energy of the bi-exciton lasing emission, in all other cases we only observe the perovskite photodegradation before reaching the condensation threshold.

To further corroborate the hypothesis that a polariton condensate has formed, we perform first-order spatial coherent measurements \( g^2(r, -r) \)—making use of a Michelson interferometer along the detection line—the Gaussian spot size is 50 × 50 μm\(^2\); (see Experimental Section for further informations). A back-reflector flips the real space emission map on one arm of the interferometer in a centrosymmetrical way so that the resulting image detected on the CCD camera is the superposition of the reference signal \( r \) and the inverted one \((-r\)\). Figure 3 shows the \( g^2(r, -r) \) signals in the linear regime (Figure 3a, at \( F = 5 \mu J/cm^2 \)) and above the second threshold (Figure 3b, at \( F = 600 \mu J/cm^2 \)). In the latter case, a typical pattern of interference is clearly visible for the whole size of the condensate, demonstrating long range coherence over 50 × 50 μm\(^2\).

Since the intensity distribution of the laser emission is highly inhomogeneous in real space (Figure S7, Supporting Information), the fringes pattern is hardly visible because of the difficulties in matching the different lasing areas.

Only high quality 2D crystals show two distinct thresholds, while we observe different behaviours studying several kind of samples with different degree of disorder. Specifically, polariton condensation does not build up when the perovskite crystals include structural defects. These defects reach the lasing threshold at very low pump power (observed also at 0.4 \( \mu J/cm^2 \) pump fluence), they are localized in space and have dispersive emission energy (Figure S8, Supporting Information). In this case, there is no observation of bi-exciton lasing or polariton condensation up to the maximum pump power before material damaging. In addition, when structural defects are present, we have also noted a reduction of the perovskite layer damaging threshold compared to the one observed in high quality 2D crystals (photodegradation can also occur for pump fluences close to 1 \( \mu J/cm^2 \)).
On the other hand, optical confinement phenomena take place at the position of some particular structural defects of the perovskite crystals,[38,39] similarly to what has been reported in 3D CsPbBr$_3$ perovskites.[40] When a defect induces a confining potential in the device plane, lasing action from multiple high-energy confined states is observed in the real space maps (Figure 4a,b) at the defect position and, as well, in the energy dispersion map (Figure 4c). Note that the lasing action from these confined states is always resonant to the defect energy band, even when varying the cavity detuning, therefore the stimulated emission can also be activated at the lowest-energy confined state (Figures S9 and S10, Supporting Information).

We have demonstrated for the first time both lasing from the bi-exciton state and the subsequent generation of a polariton condensate, in hybrid organic–inorganic layered perovskite crystals. Furthermore, to our knowledge, this is the most clear evidence of polariton condensation, in an organic or hybrid material, which is unambiguously different from lasing effect and can here be clearly distinguished. The polariton condensate forms at higher excitation power with respect to a bi-exciton lasing action. The collapse of the polariton population toward the minimum of the lower polariton dispersion turns off the bi-exciton emission and the total emission intensity abruptly increases building up a long-range spatial coherent state.

These experimental results demonstrate that such materials cannot be described using a simple two-level-system model since many energetically competitive phenomena are at stake.

This work sheds new lights on the physics of hybrid and organic crystals in optical confined systems, giving the possibility to investigate quantum coherent states based on organic–inorganic 2D layered perovskites.

**Experimental Section**

**Synthesis of 2D Perovskite Flakes:** 1 M PEAI solution was prepared in a nitrogen-filled glovebox by dissolving PbI$_2$ and phenethylammonium iodide (12 molar ratio) in γ-butyrolactone and stirring at $70^\circ$C for 1 h. 2D perovskite single crystals were synthesized using an antisolvent vapor-assisted crystallization method. 5 μL of perovskite solution were deposited on top of a sputtered DBR (or a glass substrate) and covered with a glass coverslip. These substrates and a small vial containing 2 mL of dichlorometane (antisolvent) were placed inside a bigger Teflon vial which is closed with a screw cap and left undisturbed for 12 h. After this time, millimeter-sized crystals appeared in between the two substrates and their thickness vary from few to tens of micrometers. Using SPV 224PR-M Nitto Tape or PDMS, mechanical exfoliation was carried out on the perovskite flakes in order to obtain single crystals having the desired thickness.

**Microcavity Sample Fabrication:** The DBR was made by seven pairs of TiO$_2$/SiO$_2$ (53 nm/89 nm) deposited by radio-frequency (RF) sputtering process—in an Argon atmosphere under a total pressure of $6 \times 10^3$ mbar and at RF power of 250 W—on top of a 170 μm glass substrate. The perovskite single crystals were grown on top of the DBR (see above) and a 80 nm-thick layer of silver was thermally evaporated on top of the structure (deposition parameters: current = 280 A, deposition-rate = 3 Å/s).

**Optical Measurements:** Figure 5 shows the sketch of the setup used for the optical measurements. The measurements were performed at cryogenic temperature ($T = 4$ K) using a 50-fs pulsed laser at 470 nm (10 kHz repetition rate) for emission measurements and a white light Xenon lamp for the reflectance spectra.

Through a first detection path, the image on the back focal plane of the detection objective was projected on a spectrometer entrance slit. The spectrometer was coupled to an enhanced CCD camera for the detection of the polariton energy dispersion. A second detection path was used for the spatial intensity maps and spatial coherence measurements ($g^2(r,\phi)$). In this latter case, the single-crystal emission signal entered into a Michelson interferometer; a movable retroreflector was placed along one of the two arms of the interferometer in order to invert the image in a centrosymmetrical configuration. The image containing the superposition of the two interferometer arms was then sent to a CCD camera. The overall magnification was 30 x.

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**Figure 4.** a,b) Real space emission intensity maps in presence of a 0D defect for two different pump fluences. The non-linear increase of the confined-states emission intensity demonstrate the lasing action (Section S8, Supporting Information). Gaussian excitation spot size of 50 × 50 μm$^2$. c) Energy versus momentum emission of the confined states for $F = 0.4 \mu$J/cm$^2$.

**Figure 5.** Sketch of the optical setup.
Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest
The authors declare no conflict of interest.

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