Solution-processed PbS quantum dot infrared laser with room-temperature tunable emission in the optical telecommunications window

G. L. Whitworth, M. Dalmases, N. Taghipour and G. Konstantatos

Solution-processed semiconductor lasers have achieved much success across the nanomaterial research community, including in relation to organic semiconductors\textsuperscript{1,2}, perovskites\textsuperscript{3,4} and colloidal semiconductor nanocrystals\textsuperscript{5–24}. The ease of integration with other photonic components and the potential for upscaling using emerging large-area fabrication technologies (such as roll-to-roll)\textsuperscript{25} make these lasers attractive as low-cost photonic light sources that can find use in a variety of applications, including integrated photonic circuits\textsuperscript{25–27}, telecommunications\textsuperscript{28–30}, chemo-/bio-sensing\textsuperscript{31–33}, security\textsuperscript{34} and lab-on-chip experiments\textsuperscript{35}. However, for fibre-optic or free-space optical communications and eye-safe LIDAR applications, room-temperature solution-processed lasers have remained elusive. Here, we report a solution-processed laser comprising PbS colloidal quantum dots integrated on a distributed feedback cavity, with tunable lasing wavelength from 1.55 μm to 1.65 μm. These lasers operate at room temperature and exhibit linewidths as low as ~0.9 meV.

Owing to their wavelength tunability, high quantum confinement potential and solution processability, colloidal quantum dots (CQDs) have been employed successfully in a wide variety of optoelectronic devices, such as light-emitting diodes\textsuperscript{36}, photovoltaics\textsuperscript{37–39}, lasers\textsuperscript{40–42} and photodetectors\textsuperscript{43–45}. However, demonstrations of optical gain and lasing have largely been confined to CdSe-based visible emitters due to their high gain and low degeneracy\textsuperscript{20,22,24,25,46–48}. The eightfold degeneracy of PbS(e) CQDs has limited the gain performance of Pb-salt semiconductor nanocrystals, along with the high-temperature-dependent Auger recombination rates\textsuperscript{11,25}. As a result of this, and despite optical gain in PbSe(e) being demonstrated as early as 2003\textsuperscript{28}, room-temperature lasing has yet to be reported. There has been only one report of cryogenic CQD PbS lasing, where a CQD-filled microcapillary was used to generate a whispering-gallery-mode laser with a full-width at half-maximum (FWHM) of 11 nm (ref. 28). This was performed at cryogenic temperature to inhibit Auger recombination, and room-temperature lasing could not be observed. Recently, lasing at 1.374 nm has been reported from AgSe CQDs in a microring ‘coffee stain’ resonator, but this is neither practical nor can be easily tuned\textsuperscript{49}. Although CQD lasing using industrially relevant optical cavities has been extensively reported in the visible regime\textsuperscript{50–52}, it has remained elusive for the infrared. In this Letter, we employ a distributed feedback (DFB) cavity on a highly thermally conductive substrate to achieve the first infrared CQD room-temperature, eye-safe lasing, tunable across the minimum-loss window of the telecommunications spectrum (known as the central C-bands and long-wavelength L-bands).

We go on to demonstrate a 40% reduction in lasing threshold by using a heavily n-doped PbSe CQD film\textsuperscript{53}.

Because of their low thresholds, high modal selectivity and compatibility with solution processing, a DFB architecture was chosen as the ideal, industrially relevant photonic cavity to achieve lasing in PbS CQDs\textsuperscript{20–22}. DFB lasers are waveguide gain structures in which a cavity on a highly thermally conductive substrate to achieve the first infrared CQD room-temperature, eye-safe lasing, tunable across the minimum-loss window of the telecommunications spectrum (known as the central C-bands and long-wavelength L-bands).

$$m\lambda_0 = 2n_{\text{eff}}\Lambda$$

(1)

where $\lambda_0$ is the feedback lasing wavelength, $\Lambda$ is the period of modulation and $m$ is the order of diffraction used to generate optical feedback. All the lasers presented were designed as one-dimensional (1D) second-order DFB lasers ($m = 2$). The periodic modulation in the waveguide is typically enabled by fabricating a nanostructured grating substrate and depositing the desired thickness of gain material (CQDs) to satisfy equation (1) at the desired wavelength. GRATINGS were fabricated from sapphire (Al$_2$O$_3$) substrates using electron-beam lithography and subsequent reactive-ion-etching pattern transfer, as schematically shown in Fig. 1a. Sapphire was chosen for its high thermal conductivity to avoid excessive heating in the CQDs when optically pumped, which would cause a blueshifting gain spectrum and exacerbate Auger recombination losses (Supplementary Section I). CQDs were spin-coated onto the gratings, followed by a subsequent ligand exchange in a layer-by-layer fashion (described in the Methods) to achieve the desired thickness (Fig. 1a).

To precisely design the DFB cavity to achieve lasing at the amplified spontaneous emission (ASE) peak of the PbS CQDs, a variable-gain finite-difference frequency-domain (FDFD) model was used to mimic experimental reality and predict the exact DFB lasing cavity modes\textsuperscript{16,21} (Supplementary Section II). The refractive index and extinction coefficient of films made of 5.7-nm quantum dots (absorption peak at 1,520 nm) such that

$$\bar{n}_d = n_d + i \left( k_d - A_\sigma e^{-(\lambda - \lambda_{\text{ASE}})/2\sigma_{\text{ASE}}^2} \right)$$

(2)
where \( n_i \) is the complex refractive index and \( n_r \) and \( k_r \) are the ellipsometry-measured refractive index and extinction coefficient, respectively. Figure 1c,d shows the resultant transverse electric (TE) field at 1,600 nm for a DFB structure at zero gain (\( A_g = 0 \)) and at cavity gain (\( A_g = 0.103 \)). As shown in Fig. 1c, at cavity gain, optical confinement is significantly increased, with a field enhancement factor nearly four orders of magnitude higher than that at zero gain. Figure 1f shows the simulated transmission spectrum of the structure at cavity gain, depicting a peak above unity transmission, centred at the desired lasing wavelength of 1,600 nm. The schematic representation of the fabrication process shown in Fig. 1a was then followed to create the desired simulated structure, a cross-sectional image of which is provided in Fig. 1g. From this image, we measured the grating height to be 49 nm, the period is 885 nm, the average width of the grating ridges was measured to be \( \pm 5 \) nm and the CQD layer thickness \( \sim 105 \) nm.

This relatively low value has been identified to be due to the high ground-state self-absorption of the CQDs as well as the high refractive index contrast between the CQDs and the sapphire substrate, as discussed in Supplementary Section XI. The sample was then optically pumped using a femtosecond Yt:YAG 1,030-nm laser and the DFB laser emission was collected perpendicular to the surface as described in the Methods (Fig. 2b). Figure 2c shows the collected spectrum below (black) and above (red) the lasing threshold, as well as the ASE profile (blue). Above threshold, the FWHM of the spectra drops dramatically to 3 nm (~1.5 meV), as shown in Fig. 2d. The insets in Fig. 2d show the surface of the CQD DFB above and below threshold. Below threshold, the pump stripe can be seen overlapping with a central 1-mm x 1-mm grating, along with adjacent gratings present on the sample. Above threshold, intense light emission is emitted from the grating area (Supplementary Video 1). The integrated spectral output is plotted against the optical pump fluence in Fig. 2e, giving the pump fluence lasing threshold as 930 \( \mu \)J cm\(^{-2} \). Also shown, in the inset, is the above-threshold beam profile. Assuming a packing density of 74% for the CQDs in the film and with a measured absorption of 8.7% at 1,030 nm, this pump fluence equates to an average exciton population per dot of \( N = 4.3 \), in good agreement with previously reported thresholds for optical gain and stimulated emission from this system.

By varying the DFB grating period by 5 nm, tunable lasing was achieved across the telecommunications L-band (1,565–1,625 nm).
using the same quantum dot films (diameter of 5.7 nm), producing lasing from 1,588 nm to 1,600 nm. To expand the lasing to the adjacent central C-band (1,530–1,565 nm) and the ultra-long U-band (1,625–1,675 nm), 5.4-nm and 6.0-nm CQDs were synthesized with corresponding ASE peaks at 1,558 nm and 1,648 nm, respectively. Using the previously described steps of simulations and fabrication, fully tunable lasing was achieved, traversing the telecommunications minimum-loss window from 1,553 nm to 1,649 nm, as shown in Fig. 3a. The inset in Fig. 3a shows a box plot of the FWHM of the measured laser spectra (showing an average of 1.7 ± 0.5 meV; 3.4 ± 1 nm), with some exhibiting single-mode behaviour with linewidth as narrow as 0.9 meV (<2 nm) at 1,629 nm. Although these linewidth energies compare well to other experimental infrared semiconductor DFB lasers33,34 (Supplementary Section IV), they remain broad compared to off-the-shelf, industrial-grade DFB lasers due to their low Q factors, as discussed in Supplementary Section XI. Additionally, it has been reported previously that high-carrier-density transients generated from pulsed excitation lead to a wavelength chirp effect and therefore linewidth broadening33.

Figure 3b plots the measured thresholds of the individual lasers presented in Fig. 3a, along with the ASE threshold of the respective quantum dots. We can see, in the case of L-band lasing, that the lasing thresholds are lower than that of the unstructured film ASE threshold, indicating a high degree of optical confinement and improving the radiative emission of the quantum dots in the cavity. Also observed is that, for lasing that is blueshifted compared to the ASE peak, as in the case of the C-band lasers, the threshold rapidly increases from that of the ASE, indicating a rapid fall off in gain, despite the lower self-absorption losses. Additional lasing stability and polarization measurements are provided in Supplementary Section V. Polarization measurements confirmed these lasers to be operating in the TE mode.

Following the successful demonstration of tunable lasing from intrinsic PbS quantum dots, we now demonstrate lasing in robustly n-doped PbS quantum dots. Using 1-ethyl-3-methylimidazolium iodide (EMII) in a ligand exchange solution, I− anions were exchanged with exposed S2− ions on the exposed (100) crystal plane surface of the quantum dots, as shown in Fig. 4a. Doping was achieved following a subsequent alumina atomic layer deposition (ALD) step, in which atmospheric electron acceptors (water/oxygen) were removed from the CQD surface, simultaneously encapsulating the film (as described in previous work30). This anionic exchange populates the conduction band of the CQD with electrons, reducing the number of excitons per dot needed to achieve population inversion24,30. Although doping electrons in the conduction band leads to higher Auger losses for single excitons, the reduction in gain threshold results in an overall reduction...
of Auger recombination at threshold when comparing doped and undoped CQDs. The FTIR transmission spectra of undoped and doped CQDs are provided in Fig. 4b. Clear bleaching of the exciton peak is observed, corresponding to a conduction-band occupancy of $N = 3.2$.

Following the same process as already described, a doped CQD laser (1,650 nm) was fabricated. Its input/output characteristics are shown in Fig. 4c. The doped lasing threshold was measured to be 430 $\mu$J cm$^{-2}$ of optical pump fluence. This represents a 43% reduction in lasing threshold compared to an undoped CQD laser of the same wavelength, which was measured to be 770 $\mu$J cm$^{-2}$. This matches well the ~40% occupancy of the conduction band as measured by the FTIR transmission (Methods), supporting a threshold reduction due to n-doping of the CQDs.

In conclusion, we have now presented room-temperature tunable infrared CQD lasers. This was achieved by using PbS quantum dots, which in themselves can be tuned to provide gain across the entire minimum-loss region (C- and L-bands) of the telecommunications windows. Combining these CQDs with highly thermally conductive gratings, DFB lasers were fabricated, creating lasing...
from 1,553 nm to 1,649 nm with an average FWHM of 1.7 ± 0.5 meV and achieving values as low as 0.9 meV. The design of the lasers was facilitated by an in-house-built variable-gain FDFD solver to accurately predict lasing spectra. Following this, we demonstrated a 40% lasing threshold reduction via an anionic exchange during CQD deposition to n-dope the PbS CQDs. These results demonstrate that the previously considered limitations of PbS quantum dots, such as high degeneracy and high room-temperature Auger recombination rates, do not preclude the realization of lasing. With further photonic and material improvements, these infrared, solution-processed PbS-chalcogenide DBR lasers have great potential in integrated photonics, operating at wavelengths that are ideal for fibre-optic and eye-safe free-space optical telecommunication and LIDAR applications.

Online content
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Methods

CQD synthesis. PbS CQDs were synthesized under an inert atmosphere using the hot injection method. A mixture of 0.446 g of lead (II) oxide (PbO), 50 ml of 1-octadecene (ODE) and 3.8 ml of oleic acid was heated at 100 °C under vacuum for 1 h to form the lead precursor (lead oleate). Under argon, a solution of hexamethyldisilathiane (HMS) in 3 ml of ODE was quickly injected. After 6 min of reaction, a second solution of HMS in 9 ml of ODE was injected dropwise, then the solution was cooled naturally to room temperature. PbS CQDs were precipitated with the addition of a mixture of acetone and ethanol and redispersed in anhydrous toluene. This purification process was repeated twice more. Finally, the concentration of PbS CQDs was adjusted to 30 mg ml⁻¹ and the solution was bubbled with N₂ to avoid oxygenation. The nanocrystal size was tuned by varying the amount of HMS used in each solution. PbO, oleic acid and HMS were purchased from Sigma Aldrich. ODE was purchased from Alfa Aesar. Acetone, ethanol and anhydrous toluene were purchased from Scharlab. All reagents and solvents were used as received. CQD diameters were calculated using an empirical quadratic formula.

Grating fabrication and characterization. Gratings were fabricated in a cleanroom by spin-coating polymethyl methacrylate (PMMA; AR-P 662.04 Allresist) at 4,000 r.p.m. for 60 s on top of sapphire substrates (Ossila), followed by a 2- min baking step at 150 °C. A conductive polymer layer (AR-PC 5090.02 Allresist) was then spin-coated on top at 2,000 r.p.m., then baked at 90 °C. Samples were transferred to an electron-beam lithography system (Crestec CABL 9000C) for patterning. After lithography, the conductive polymer layer was dissolved off in water for 60 s and the electron-beam resist was developed for 2 min. After development, reactive ion etching was performed using 80 s.c.c.m. of Ar and 20 s.c.c.m. of CHF₃ under a radiofrequency power of 290 V for 5 min. The residual PMMA was then removed using an oxygen plasma asher followed by acetone/isopropanol cleaning. Focused-ion-beam milling was performed using a Zeiss Auriga system and a 100-nm gold layer was deposited on the DFB sample before using a thermal evaporator (Nano 36 Kurt J. Lesker) at a base pressure of 10⁻¹⁰ mbar.

CQD deposition. Quantum dots were deposited on alumina gratings with the following steps: (1) a 30 mg ml⁻¹ solution of CQDs was spun at 2,500 r.p.m. on gratings for 30 s; (2) a ligand exchange solution of 7 mg ml⁻¹ EMII in methanol + 0.01% MPA was applied to the surface for 30 s; (3) samples were spun at 2,500 r.p.m. for 60 s while methanol was dropcast onto the surface to clean the substrate of unwanted ligands; (4) steps 1 to 3 were then repeated until the desired thickness of CQDs was reached. For doping, the samples were subsequently transferred to an ALD machine for 30 cycles of alumina deposition.

Optical characterization. FTIR spectra were obtained using a Nicolet iS50R FT-IR spectrometer. Conduction-band occupancy, N, was calculated by taking the ratio of the exciton absorption area before and after doping. Ellipsometry was performed using a SOPRA GES-5E spectroscopic ellipsometer and the CQDs were modelled using a Tauc–Lorentz model with additional Gaussian oscillators for the primary exciton transition and additional higher-energy transitions.

ASE and lasing characterization. Samples were optically pumped using a 1,030-nm, femtosecond Yt:YAG ORIGAMI laser (NKT Photonics) at a repetition rate of 50kHz. Pulses were 300 fs long and were focused onto the samples using a cylindrical lens to a stripe of ~1.2 mm x 0.2 mm. ASE spectra were collected from the side of each sample using an f = 50 mm, two-inch lens and free-space-coupled into a Kymera 326i spectograph (Oxford Instruments, Andor) via an f = 200 mm lens through a 100-μm slit. Additional gain and loss measurements were taken from unstructured PbS CQD films (Supplementary Section VII). Lasing spectra were collected perpendicular to the surface, ~20 cm away from the DFB grating surface, using a fibre-coupled port of the spectrometer and with a 10-μm slit to obtain high resolution. All ASE and lasing spectra were collected using an integration time of 1 s, except for the low-resolution below-threshold spectra, for which 5 s was used. The experimental configurations for both ASE and lasing are provided in Supplementary Section VII. Beam profiles were taken using an NTT-WiDiy-Sens-320V-ST InGaAs camera (Iberoptics Sistemas Ópticos), ~2 cm away from the sample surface. For imaging of sample surfaces, a short-wave infrared lens was attached to the camera and the camera was placed 20 cm away from the sample. A long-pass filter was used in all situations to block the pump laser light. Spectral data and threshold calculations for all lasers presented are provided in Supplementary Section IX.

FDFD simulations. A 2D FDFD electromagnetic solver code was written in MATLAB. Periodic boundaries were applied in the x direction and perfectly matched layers were used in the y direction. The width of the simulation was set equal to the period of the grating, with the y direction equal to the height of the target structure plus a full wavelength above and below. The code was run iteratively over the structural parameters (for example, height and period), wavelength and gain amplitude, with the refractive index being adjusted accordingly for each iteration. The refractive indices for alumina were obtained from refractivindex.info (https://refractivindex.info/). The field enhancement factor (Γ) was calculated along the central axis according to the following equation:

\[
FEF = \frac{\int |E(z)|^2 dz}{\int |E_{source}|^2 dz} \tag{3}
\]

where \(E\) denotes the resultant out-of-plane electric field from the FDFD simulations and \(E_{source}\) is the source field.

Data availability

The data presented in this study are available in Zenodo with the identifier https://doi.org/10.5281/zenodo.5112914. Supplementary Information data are available from the corresponding author upon reasonable request.

Code availability

FDFD MATLAB codes are available in Zenodo with the identifier https://doi.org/10.5281/zenodo.5112914.

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Author contributions

G.K. designed, supervised and directed the study. G.L.W. designed the experiments, and the thin films. M.D. synthesized the quantum dot materials. N.T. carried out FIB and SEM characterization measurements and contributed to thin film and laser development. G.L.W. and G.K. wrote the manuscript, with input from the co-authors.

Competing interests

The authors declare no competing interests.

Additional information

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Correspondence and requests for materials should be addressed to G. Konstantatos.

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