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SUMMARY

Colloidal quantum dots (CQDs) and other semiconductor (SC) nanocrystals (NCs), like perovskites, are nanosized semiconductor particles with a tunable bandgap and interesting optical properties. CQD and other SC NCs, have been an active field of research for several decades because of their versatility in terms of synthesis and application. As solution-processable, tunable semiconductor, CQDs and NCs have been envisioned for numerous applications ranging from active material and stabilizer in photovoltaics, spectral downshifting for solar and display technologies, photodetectors and lasers, among others, some of which are already commercial mass products. The tunability of NCs makes it relatively easy to optimize the material to the intended application in terms of bandgap, absorption, emission wavelength, conductivity, charge transfer and compatibility with different surrounding materials and chemical environments. At the same time, patterning these nanocrystals at the nanoscale could add another knob to turn in order to expand the usability of CQDs or improve their performance. In this thesis we present work on patterning of CQDs and NCs with several lithographic techniques.

Chapter 1 starts with an introduction to quantum dots, nanocrystals and lithography. We cover the basics of the synthesis of II-VI CQDs as well as perovskite NCs. We introduce some of their applications and provide insight in the current landscape of patterned NCs. Next to this, we cover some fundamentals of lithography, both in the form of deep-ultraviolet lithography (DUVL) and extreme-ultraviolet lithography (EUVL) as well as electron-beam lithography (EBL).

In **Chapter 2** we demonstrate a general one-step nanopatterning technique for as-synthesized PbS and CdSe CQDs with advanced photolithography. We find we can use both relatively low energy photons (from 5.5 eV), as well as higher energy EUV photons (91.9 eV) for patterning features as small as 60 nm directly.

We find that the solubility change is achieved by cross-linking the organic ligands, to create a cohesive CQD film. Required doses are relatively low and, in the order of commercial state-of-the-art patterning materials ($\sim 120 \text{ mJ cm}^{-2}$) for EUV. The exposure to EUV photons, even to relatively high doses, does not significantly affect the luminescent properties, which allows for fabrication of luminescent structures directly.

In **Chapter 3** we expand on our patterning of PbS and CdSe CQDs with e-beam-based lithography techniques as well. We find that low-energy electrons (from 3 eV) can already be used to induce insolubility reactions to pattern CQDs, yet with high electron beam dose. Highly-energetic electrons (50 keV) from commercial EBL systems allow for patterning at much lower doses. Doses on the order of several hundred $\mu\text{C cm}^{-2}$ are sufficient to induce the patterning mechanism. This dose is in the same range as commercial state-of-the-art e-beam resists. From time correlated single photon counting (TCSPC) and photoluminescence (PL) experiments we find that luminescent properties seem to be relatively unaffected by the exposure. The intricate designs that are unlocked by EBL finally allows us to create a luminescent microscale version of the famous painting *Girl with the Pearl Earring* by Johannes Vermeer.

Chapter 4 covers another extension of the technique in terms of a different materials class: perovskites. We pattern CsPbBr₃ NCs via EBL into different designs. We find it is possible to create features around 100 nm and we can pattern the nanocrystals with high precision in elaborate designs. We observe a reduction of photoluminescence quantum yield (PLQY) of the material after processing, but luminescence is nonetheless bright and radiative decay is relatively stable. We attribute the patterning mechanism to cross-linking of the surface ligands as elucidated by Fourier transform infrared spectroscopy (FTIR). The amine ligands, however, seem to partially detach from the crystal, thereby reducing the passivation.

In the final **Chapter 5** we use our patterning technique for a light emission application. We fabricate waveguiding structures

patterned from emitters directly. The direct EBL described in **Chapter 2** is a suitable fabrication technique to pattern colloidal CQDs into optical surfaces, like Bragg gratings, to manipulate light emission. We show control over the angle of emission and that a standing wave for out-of-plane emission forms in the CQD grating. We show that the patterned film can be charged but so far no lasing was observed. Optical simulations show that in our system losses are currently too high. We need to adjust our system further, for instance with index engineering, as the current structure does not confine the optical mode enough inside the emitter.

SAMENVATTING

Colloïdale kwantumdeeltjes (CKD's) en andere halfgeleider nanokristallen (NK's), zoals perovskieten, zijn halfgeleider deeltjes op nanoschaal met een aan te passen bandkloof en interessante optische eigenschappen. CKD's en halfgeleider NK's vormen een actief onderzoeksveld voor meerdere decennia door hun veelzijdigheid op gebied van synthese en toepassingen. Door de eenvoudige verwerkbaarheid in oplossing en de aanpasbaarheid van deze halfgeleiders, hebben kwantumdeeltjes en NK's talloze toepassingen gevonden, variërend van actieve materialen of stabilisator in fotovoltaïsche cellen, materiaal voor spectrale verschuiving voor zonnecel- en displaytechnologiën, fotodetectoren en lasers. De eerdergenoemde aanpasbaarheid van NK's maakt het relatief eenvoudig om het materiaal te optimaliseren voor de voorgenomen toepassing op het gebied van de bandkloof, absorptie, emissiegolflengte, geleidbaarheid, ladingsoverdracht en compatibiliteit met verschillende materialen en chemische omgevingen. Tegelijkertijd kan het vormen van patronen uit deze kristallen op nanoschaal een nieuwe knop toevoegen om aan te draaien ter uitbreiding van de functionaliteit of de toename van de prestaties van CKD's. In dit proefschrift presenteren wij werk op het gebied van het vormen van patronen uit CKD's en NK's met verschillende lithografische technieken.

Hoofdstuk 1 begint met een introductie over kwantumdeeltjes, nanokristallen en lithografie. We beschrijven de basis van de synthese van II-VI CKD's, evenals perovskiet NK's. We introduceren enkele toepassingen en geven inzicht in het huidige landschap van het vormen van patronen uit NK's. Hiernaast beschrijven we enkele fundamentele principes van lithografie, zowel in de vorm van optische diep-ultraviolet (DUV)-lithografie en extreem-ultraviolet (EUV)-lithografie als elektronenbundellithografie (EBL).

In **Hoofdstuk 2** demonstreren we een algemene techniek voor het creëren van nanopatronen uit eenvoudig gesynthetiseerde PbS en CdSe kwantumdeeltjes in een enkele productiestap met geavanceerde fotolithografie. We vinden dat we zowel fotonen met een relatief lage energie (vanaf 5.5 eV), als hoger energetische EUV-fotonen (91.9 eV) kunnen gebruiken om structuren te fabriceren die zo klein kunnen zijn als 60 nm. We vinden dat er een wisseling in oplosbaarheid plaatsvindt door vernetting van de organische liganden, waarmee een samenhangende KD-laag wordt gevormd. De benodigde doses zijn relatief laag en van dezelfde orde grootte ($\sim 120 \text{ mJ cm}^{-2}$) als moderne commerciële materialen die zijn ontworpen voor het creëren van nanopatronen. De blootstelling aan EUV-fotonen beïnvloedt de luminescente eigenschappen van de materialen niet significant, zelfs niet bij hoge doses, waardoor het mogelijk wordt om op een directe manier luminiscente structuren te maken.

In **Hoofdstuk 3** breiden we het vormen van patronen uit PbS and CdSe CKD's verder uit met elektronenbundellithografie-technieken. We vinden dat laag-energetische elektronen (vanaf 3 eV) al gebruikt kunnen worden om reacties te veroorzaken die de oplosbaarheid van de NK's verandert. Bij het gebruik van laag-energetische elektronen zijn echter hoge elektron doses vereist. Het mechanisme voor patroonvorming wordt ook geïnduceerd door hoog-energetische elektronen (50 keV) van commerciële elektronenbundellithographiesystemen. In dit geval volstaan veel lagere doses, in de orde van enkele honderden $\mu\text{C cm}^{-2}$. Dit is vergelijkbaar met commerciële resistmaterialen. Uit tijdopgelosten stabiele-toestandfotoluminescentie-experimenten vinden we dat de luminescente eigenschappen relatief ongemoeid blijven door de blootstelling aan de elektronenbundel. Omdat EBL het fabriceren van zeer ingewikkelde ontwerpen ontsluit, is het mogelijk om een luminescente microversie te fabriceren van het beroemde schilderij *Het Meisje met de Parel* van Johannes Vermeer.

Hoofdstuk 4 omvat wederom een uitbreiding op onze techniek, door het gebruik van een nieuwe klasse materialen: perovskieten. We vormen patronen uit CsPbBr_3 NK's met EBL in verschillende ontwerpen. Het is mogelijk om structuren te maken zo klein als

100 nm en kunnen deze structuren met grote precisie plaatsen, waardoor we ingewikkelde ontwerpen kunnen fabriceren. We observeren een afname in de fotoluminescentiekwantumopbrengst van het materiaal na het fabricageproces, maar luminescentie is niettemin helder en stralend verval is relatief stabiel. We schrijven het mechanisme voor patroonvorming toe aan vernetting van de liganden, zoals Fourier Transformatie Infrarood Spectroscopie aan het licht brengt. De amineliganden lijken ook deels van de kristaloppervlakte gescheiden, waardoor de passivatie van de kristallen afneemt.

In het laatste **Hoofdstuk 5** passen we onze patroonfabricage toe om lichtemissie te beïnvloeden. We fabriceren een lichtgeleidende structuur van een emitterend materiaal zelf. De directe EBL-methode die is beschreven in **Hoofdstuk 2** is een geschikt procedé voor het vormen van CKD's in optische structuren, zoals Braggroosters, om lichtemissie mee te manipuleren. We laten zien dat we controle hebben over de emissiehoek van de lichtbundel en dat een staande golf wordt gevormd die zorgt voor een directionele bundel loodrecht op de structuur. Het materiaal kan ook elektrochemisch worden opgeladen om de drempel voor optische versterking te verlagen, maar tot op heden zien we geen lasing. Optische simulaties laten zien dat er in ons huidige systeem teveel verliezen zijn. Het systeem zou verder geoptimaliseerd kunnen worden, bijvoorbeeld door indexoptimalisatie, om de optische modus beter in het emitterende materiaal te begrenzen.

LIST OF PUBLICATIONS

The chapters of this thesis are based on the following publications:

- [1] **Dieleman, C. D.**, Ding, W., Wu, L., Thakur, N., Bepalov, I., Daiber, B., Ekinici, Y., Castellanos, S. & Ehrler, B. "Universal direct patterning of colloidal quantum dots by (extreme) ultraviolet and electron beam lithography", *Nanoscale* **2020** 12(20), 11306-11316
- [2] **Dieleman, C. D.**, van der Burgt, J., Garnett, E.C. & Ehrler, B. "Direct patterning of CsPbBr₃ nanocrystals with electron-beam lithography" (*in preparation*)
- [3] **Dieleman, C. D.**, Kolkowski, R., Pal, D., Geuchies, J.J., van der Burgt, J., Garnett, E.C., Houtepen, A.J., Koenderink, A.F. & Ehrler, B. "Directly patterned, electrochemically charged colloidal quantum dots for lasing devices" (*in preparation*)

Other publications by the author:

- [4] van der Burgt, J. S., **Dieleman, C. D.**, Johlin, E., Geuchies, J. J., Houtepen, A. J., Ehrler, B., & Garnett, E. C. "Integrating sphere Fourier microscopy of highly directional emission", *ACS Photonics* **2021** 8(4), 1143-1151
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