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List of publications

This thesis is based on the following publications:

1. R. Limpens*, **A. Lesage***, M. Fujii, and T. Gregorkiewicz, *Size Confinement of Si Nanocrystals in Multinanolayer Structures*, Scientific Reports, 5, 17289 (**2015**). Chapter 2.
2. A. Capretti, **A. Lesage**, and T. Gregorkiewicz, *Integrating Quantum Dots and Dielectric Mie Resonators: A Hierarchical Metamaterial Inheriting the Best of Both*, ACS Photonics, 4(9), 2187-2196 (**2017**). Chapter 3.
3. **A. Lesage**, D. Timmerman, D.M. Lebrun, Y. Fujiwara, and T. Gregorkiewicz, *Hot-Carrier-Mediated Impact Excitation of Er^{3+} Ions in SiO_2 Sensitized by Si Nanocrystals*, Applied Physics Letters, 113(3), 031109 (**2018**). Chapter 5.
4. **A. Lesage**, D. Timmerman, T. Inaba, T. Gregorkiewicz, and Y. Fujiwara, *Enhanced Light Extraction Efficiency of Eu-Related Emission from a Nano-Patterned GaN Layer Grown by MOCVD*, Scientific reports, 9(1), 4231 (**2019**). Chapter 4.

Under preparation:

5. **A. Lesage**, M. van der Laan, L. Gomez, and T. Gregorkiewicz, *Doping of Yb^{3+} in $CsPbBr_xCl_{3-x}$ nanocrystals*. Chapter 6.
6. **A. Lesage**, B. Smits, and T. Gregorkiewicz, *Acceptors and donors in energy transfer between Si NCs*. Chapter 7.
7. **A. Lesage**, A. P. Westerveld, S. ten Have, L. Gomez, and T. Gregorkiewicz, *Emission mechanisms in $CsPbX_3$ nanocrystals studied by photoluminescence spectroscopy*. Chapter 8.

Other publications by the author:

8. R. Limpens, **A. Lesage**, P. Stallinga, A.N. Poddubny, M. Fujii, and T. Gregorkiewicz, *Resonant Energy Transfer in Si Nanocrystal Solids*, The Journal of Physical Chemistry C, 119(33), 19565-19570 (**2015**).
9. N.X. Chung, R. Limpens, **A. Lesage**, M. Fujii, and T. Gregorkiewicz, *Optical Generation of Electron–Hole Pairs in Phosphor and Boron Co-Doped Si Nanocrystals in* SiO_2, physica status solidi (a), 213(11), 2863-2866 (**2016**).
10. J. Takatsu, R. Fuji, J. Tatebayashi, D. Timmerman, **A. Lesage**, T. Gregorkiewicz, and Y. Fujiwara, *Growth and Optical Characteristics of Tm-Doped AlGaN Layer Grown by Organometallic Vapor Phase Epitaxy*, Journal of Applied Physics, 123(16), 161406 (**2018**).
11. N.X. Chung, R. Limpens, C. de Weerd, **A. Lesage**, M. Fujii, and T. Gregorkiewicz, *Toward Practical Carrier Multiplication: Donor/Acceptor Codoped Si Nanocrystals in* SiO_2, ACS Photonics, 5(7), 2843-2849 (**2018**).

Summary

Controlling Light Emission of Nanoparticles

In this PhD thesis, titled ”Controlling Light Emission of Nanoparticles” we try to improve our understanding of how nanostructures behave and how we can manipulate their light emission. The light we observe from these materials originates from the nanoparticles within them: nanocrystals and rare-earth ions. In this study, different aspects within the photoluminescence process of the nanoparticles are addressed in order to ultimately make them more attractive for application.

We employ two different methodologies to study and influence the nanomaterial properties. Firstly, in chapters 2-4, we manipulate the surrounding of the emitters, while keeping the inherent carrier properties within the nanoparticles unperturbed. Secondly, in chapters 5-8, we focus on the carrier properties, and attempt to understand and influence different radiative and non-radiative mechanisms related to photoluminescence. What then follows is a breakdown of each of these parts into the constituent chapters.

In chapter 2 the structure in which the nanoparticles – silicon nanocrystals (Si NCs) – are embedded, is modified. We focus on a particular fabrication technique: high temperature annealing of sputter-deposited Si-rich SiO_2 (SiO_x), and explore the use of multilayer (ML) structures as an approach to effectively control the NC size. We find that ML structures do give more control over size, but this is not by the layer thickness, but by diffusion of silicon inside the Si-rich nanolayers and across the SiO_2 spacer layers. This insight allows us to better understand how to fabricate better Si NCs.

Next we try to address a fundamental issue of Si NCs: their broad light absorption. In order to use Si NCs as efficient downconverters, we want to make use of their efficiency enhancing mechanisms which can only be applied in a selected part of the solar spectrum. But Si NCs have broad light absorption, and while they might enhance one part, they also absorb other parts of the solar spectrum, but at a loss. In chapter 3 we attempt to taylor the absorption by the Si NCs, to those parts of the solar spectrum we can utilize efficiently. By employing a metamaterial structure that gives rise to Mie resonances, we can effectively focus only a part of the solar spectrum onto the NCs. We find that even though we have less Si NCs in the metamaterial than in the reference sample, their selective light absorption and resulting emission is increased. Such a metamaterial structure can easily be used in future devices.

In analogy to the former chapter, in chapter 4 we again make use of a metamaterial. This time not to enhance selective absorption and resulting emission, but to enhance only the light emission itself. Red emission of Eu-doped GaN (GaN:Eu) is highly desirable and has many applications in light emitting devices. The problem is that current GaN:Eu materials show insufficient emission intensities. The metamaterial explored here enhances the extraction efficiency of red Eu emission out of the GaN layer, and directional light output is increased by 60%. We even find that per available Eu-emitter in the material, the directional light output has increased by up to 12.8 times.

Having explored three different material structures that enable different kinds of control over the light emission properties, we now proceed to explore properties that relate to the mechanisms and processes happening within and between nanoparticles. In both chapter 5 and chapter 6, we make use and explore sensitization mechanism of rare-earth (RE) ions. Sensitization proceeds through energy transfer from photo-generated carriers in the hosting material to the RE ion, promoting an excitation of the ion. Effectively, we excite the RE ions by exciting the hosting material, and so if the host is easily excited, this could translate to efficient sensitization. ”Could” – because the transfer from host to ion is not easy, and in each of the two chapters we explore the mechanism of this transfer in two different systems.

Erbium (Er) ions codoped into the SiO_2 host together with Si NCs, can be excited by two different transfer processes, a ”slow” and ”fast” one. In chapter 5 we explore the ”fast” one, as it is potentially more efficient, and show that it is mediated by impact-excitation. The sensitizing Si NC needs to be excited with sufficient energy: either by absorption of a single energetic photon, or of multiple small energy photons which then proceed to recombine into a single high energy exciton by an Auger process. Both of these cases result in fast energy transfer to the Er ion.

Perovskite NCs are a natural candidate for dopant sensitization owing to their high tolerance to crystal defects and superb optical properties. In chapter 6, $CsPbX_3$ NCs are employed as sensitizers for Yb ions. We observe not only efficient sensitization, but also find that sensitization requires the use of $CsPbCl_3$. The larger bandgap of $CsPbCl_3$ couples and excites two Yb ions at once. Moreover, we find no indications of impact-excitation that would enable higher efficiency but speculate on how it could be achieved in the future with perovskite NCs of different composition.

In chapter 7 we return to Si NCs, and now we explore the energy transfer process between NCs. Energy transfer is assumed to be prevalent in Si NCs, allowing excitons to transfer from NC to NC, and possibly also to dark NCs, in that way decreasing the average emission efficiency. We employ a technique in which we look at the NCs’ emission at low temperature in two cases: in one case allowing them to transfer freely, and in the other by populating the neighboring acceptor NCs which quench any transferred exciton. This allows us to probe the acceptor NCs, and we show that, contrary to expectation, not only large NCs, but also small NCs can be on the receiving end of energy transfer. This might have big implications for the so-called excitonic solar-cells which rely on diffusion of either energy or carriers as a way to harvest the absorbed solar energy.

Lastly, in chapter 8, we explore the emission properties of $CsPbX_3$ perovskite NCs. While widely investigated, the emission mechanism of $CsPbX_3$ perovskite NCs is still not completely understood. We start with the simpler low temperature emission properties and extend the understanding towards the thermally dominated room temperature processes. We identified the signature of dark NCs at room temperature, and speculate on their different contributions to the observed emission spectra. Among these we consider polaron formation, phase changes, and the influence of space quantization. While we do not provide a complete understanding, we do propose a framework in which to consider the emission properties of these materials in the future.

Samenvatting

Beheersing van lichtemissie van Nanodeeltjes

In dit proefschrift wordt getracht meer inzicht te krijgen in hoe een selectie nanomaterialen zich gedraagt. Daarnaast wordt onderzocht hoe invloed kan worden uitgeoefend op de lichtemissie eigenschappen van deze materialen en vervolgens hoe deze benut kunnen worden. Het licht afkomstig van silicium, perovskiet en galliumnitride ontstaat uit de nano-deeltjes waaruit ze zijn opgebouwd: nano-kristallen en zeldzame aardmetalen. Verschillende aspecten van het lichtemissie-proces van de nano-deeltjes komen aan bod met het doel ze te kunnen toepassen in toekomstige technologische ontwikkelingen.

Er wordt gebruik gemaakt van twee methodes om de eigenschappen van de nanodeeltjes te beïnvloeden. Ten eerste, in hoofdstukken 2-4, veranderen we de omgeving van de nano-deeltjes terwijl we geen directe invloed uitoefenen op de ladingdragers in de deeltjes (de elektronen bijvoorbeeld). Vervolgens, in hoofdstukken 5-8, leggen we juist de focus op de ladingdragers die verantwoordelijk zijn voor de processen in en tussen de nano-deeltjes. Hierna volgt een uitvoerige uitleg van elk van de hoofdstukken.

In hoofdstuk 2 bestuderen we de structuur waarin silicium (Si) nano-kristallen zijn omgeven. We leggen de focus op een bepaalde fabricagetechniek (kristalgroeitechniek) van de nano-kristallen: sputter-depositie van laagjes siliciumrijk siliciumdioxide, gevolgd door hoge temperatuur tempering. Hierbij maken we gebruik van een multilaag-structuur opgebouwd uit laagjes die nano-kristallen bevatten en daartussen barrière-lagen. Deze structuur moet ervoor zorgen dat de grootte van de nanokristallen beperkt wordt door de dikte van de laagjes waarin de kristallen groeien. Maar wat blijkt, de kristalgroei wordt niet beperkt door de laagdikte, echter de dikte van de tussenliggende barrière-lagen bepaalt of groeibeperking zal plaatsvinden. Met deze nieuwe kennis kunnen nano-kristallen met een nauwkeurig gedefinieerde grootte gefabriceerd worden, en daarmee ook de eigenschappen van verschillende diameters beter worden onderscheiden.

Vervolgens wordt de focus gelegd op een fundamenteel probleem van Si nanokristallen: de brede band van lichtabsorptie. Als we Si nano-kristallen een toepassing willen geven als licht-omvormer voor zonnecellen, dan is het nodig om gebruik te kunnen maken van de unieke efficiëntie-verhogende processen die de nano-kristallen bezitten. Deze efficiëntie-verhogende processen vinden plaats in een beperkte band van frequenties in het hele zonnespectrum, maar de nano-kristallen absorberen meer frequenties dan die beperkte band. Het doel in hoofdstuk 3 is om de absorptie te verhogen voor een bepaalde frequentieband, en verlagen voor de rest. Op die manier proberen we het materiaal toepasbaar te maken voor allerlei technische applicaties. Door de nano-kristallen te fabriceren in een bepaalde structuur die een meta-materiaal wordt genoemd, ofwel een periodieke structuur met een golflengte kleiner dan het licht, ontstaan er Mie resonanties. Deze Mie resonanties zorgen voor een verhoogde selectieve absorptie van bepaalde frequenties.

In hoofdstuk 4 maken we op een soortgelijke wijze gebruik van een meta-materiaal maar wel met enkele essentiële verschillen. In dit geval gaat het niet om invloed uit te oefenen op de lichtabsorptie en de resulterende emissie, maar zal enkel de lichtemissie beïnvloed worden. We maken hier gebruik van gallium-nitride (GaN) waarin het zeldzame aardmetaal europium (Eu) is verwerkt, met als resultaat Eu gedoteerde GaN (Eu:GaN). GaN is een veel gebruikte halfgeleider voor LEDs, en de toevoeging van Eu geeft de mogelijkheid om rood-licht LEDs op basis van GaN te realiseren. Het probleem met huidige Eu:GaN materialen is de te lage rood-licht emissie-intensiteit. Het meta-materiaal dat hier wordt onderzocht, verhoogt de extractie van lichtemissie uit de Eu:GaN. Het zorgt voor directioneel licht met een intensiteit die tot wel 60

Na het onderzoeken van de omgeving van de nano-deeltjes om de emissie ervan te beïnvloeden, zal er nu ingezoomd worden naar welke processen zich binnenin en tussen de nano-deeltjes plaatsvinden, en hoe deze zich manifesteren in de emissie eigenschappen. In de hoofdstukken 5 en 6, bestuderen we de excitatie mechanismen van zeldzame aardmetalen (ZA's) verwerkt in Si nano-kristallen en perovskiet nano-kristallen. ZA's zijn normaliter moeilijk te exciteren, maar door gebruik te maken van energie-overdracht van nano-kristallen op ZA ionen, is excitatie wel mogelijk. Dit overdrachtsproces vind niet gemakkelijk plaats omdat het afhankelijk is van verschillende eigenschappen van de nano-kristal. In dit proefschrift zullen de verschillende overdrachtsmechanismen worden onderzocht en vervolgens zal er gekeken worden hoe we de overdracht kunnen faciliteren. Net zoals Eu in GaN, bieden deze ZA's kansen voor vele toepassingen. Erbium en ytterbium, beide ZA's, worden veel gebruikt voor hun optische eigenschappen in lasers en glasvezels. Het faciliteren van een efficiëntere excitatie met behulp van nano-kristallen is daarom wenselijk.

Erbium (Er) ionen die samen met Si nano-kristallen worden verwerkt in siliciumdioxide (SiO_2), kunnen geëxciteerd worden door de nano-kristallen. Eerder zijn er twee mogelijke overdrachtsprocessen tussen de nano-kristal en de Er ion geïdentificeerd, een langzaam en een snel proces. In hoofdstuk 5 bestuderen we het snelle proces, omdat het onderliggende mechanisme nog onbekend was en omdat het proces potentieel veel efficiënter is. Uit dit onderzoek is gebleken dat een proces genaamd impact-excitatie ten grondslag ligt aan het snelle proces. Om impact-excitatie te faciliteren is het nodig dat de nano-kristal voldoende energie krijgt zodat er een exces aan energie in de nano-kristal ontstaat. De energie-exces kan dan worden overgedragen aan een Er ion, en op die manier gaat die exces niet verloren. Dit proces van energie overdracht van enkel het energie-exces noemen we impact-excitatie. Om de nano-kristal een energie-exces te geven kan op twee manieren worden bereikt, met een enkele hoge energie foton of met meerdere lage energie fotonen die in de nano-kristal samen recombineren tot een hoge energie excitatie (Auger recombinatie). Bij afwezigheid van deze exces energie zien we het snelle proces verdwijnen, en blijft alleen het langzame proces optreden. Hiermee is de onderliggende mechanisme van de snelle overdracht proces geïdentificeerd.

De opname van ZA ion in een bestaande kristalstructuur kan deze structuur verstoren, en daarmee negatieve effecten veroorzaken. Perovskiet nano-kristallen hebben bewezen zeer tolerant te zijn tegen deze structuur-storingen – kristal-defecten. Die tolerantie alsmede de uitstekende optische eigenschappen maken perovskiet nano-kristallen natuurlijke kandidaten om ZA's te exciteren. In hoofdstuk 6 worden $CsPbX_3$ nano-kristallen gebruikt om ytterbium (Yb) te exciteren. We vinden niet alleen zeer efficiënte excitatie van de Yb ion door de nano-kristallen we verder hoe we dan wel de excitatie efficiëntie verder kunnen verhogen.

Hoofdstuk 7 bestudeert weer Si nano-kristallen, en hierbij wordt gekeken naar de overdracht van energie tussen verschillende nano-kristallen. Energie-overdracht (EO) wordt gezien als een gangbaar proces, waarbij de excitatie energie – de exciton – wordt overgedragen van nano-kristal naar nano-kristal. We weten al dat er ook defecte — donkere nano-kristallen bestaan, en het is waarschijnlijk dat EO naar een donkere kristal ook bijdraagt aan de energieverliezen. EO wordt onderzocht door de ontvangers van EO te beïnvloeden. Als deze al geëxciteerd zijn dan zal een EO naar een "bezette" kristal tot niets leiden, en dit wordt herkend door de afname van lichtemissie. Op die manier wordt geprobeerd de ontvangers van EO te identificeren. In tegenstelling tot wat er van tevoren werd gedacht, functioneren niet alleen grote nano-kristallen als ontvangers maar ook de kleinere nano-kristallen. Dit kan grote gevolgen hebben voor de zogenoemde excitonische zonnecellen waarbij er gebruik wordt gemaakt van energie-overdracht om de energie uit de zonnecel te extraheren.

Ten slotte, hoofdstuk 8 bestudeerd de emissie eigenschappen van $CsPbX_3$ perovskiet nano-kristallen. Terwijl deze groep materialen al enkele jaren veel worden bestudeerd, is het lichtemissie-proces niet compleet duidelijk. Eerst wordt er gekeken naar de lage temperatuur eigenschappen, om vervolgens de processen die bij lage temperatuur worden geïdentificeerd uit te breiden, en de complicaties die kamertemperatuur met zich mee brengt mee te nemen in die processen. Donkere kristallen tussen de nano-kristallen worden ook geïdentificeerd bij kamertemperatuur. Vervolgens worden allerlei mogelijkheden overwogen om de emissie-eigenschappen te verklaren. Onder meer overwegen we Polaron formatie, fase overgangen en de invloed van ruimte kwantisatie. Ook al hebben we geen compleet begrip vergaard, zijn er wel verschillende kaders geschetst waarin de emissie-eigenschappen in de toekomst bestudeerd kunnen worden.

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