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Photophysics of Metal Halide Perovskites

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Summary

Global warming exerts pressure to enhance the implementation of renewable energy sources by both private and public stakeholders. Solar photovoltaic (PV) technology, characterised by its widespread availability and cost-effective production, will become even more important in the following years. However, the existing PV technology based on silicon faces limitations in its application. In contrast, emerging PV technology holds the potential for deployment in buildings and on warehouse rooftops, as well as for surpassing the efficiency of PV panels solely reliant on silicon.

Metal halide perovskites are at the centre of attention in new PV technology, exhibiting an impressive increase in solar cell efficiency to over 25% in the past decade. However, there is growing concern about the stability and toxicity of the most commonly employed Pb-based perovskites. Strategies to mitigate these concerns are incorporating large organic cations for improved ambient stability and structural flexibility, alongside the substitution of Pb with the more environmentally benign Sn. While these materials have found successful applications in solar cells and LEDs, many of their fundamental properties are subject to debate or are unknown. This thesis addresses the photophysics of an extensive range of compounds, unravelling their properties and contributing to a deeper understanding that facilitates the rational design of new materials.

In Chapter 3 we investigated $(\text{PEA})_2\text{PbI}_4$ and $(3\text{-FPEA})_2\text{PbI}_4$ single crystals, which exhibited both a narrow emission in the green and a broad emission in the red. The broad emission has been observed recently in a number of perovskite-related compounds, and its origin has been mostly attributed to self-trapped excitons. Instead, we showed that the broad emission originates from defects in the band gap, hereby addressing a common misconception in the scientific community. A crucial, yet simple, below-gap excitation experiment allowed us to make this distinction.

We continued our investigation on $(\text{PEA})_2\text{PbI}_4$ and $(3\text{-FPEA})_2\text{PbI}_4$ in Chapter 4 by studying the narrow emission band through a high-resolution temperature-dependent photoluminescence study. The emission on the high-energy side of these compounds is largely affected by the choice of the organic cation, where the separation of the hot-exciton resonances is reduced from ≈ 40 meV to ≈ 15 meV for $(\text{PEA})_2\text{PbI}_4$ and $(3\text{-FPEA})_2\text{PbI}_4$, respectively. In contrast, emission from the low-energy side of the free-exciton emission stems from excitons bound to defects, as revealed by their time-resolved decays and distinct temperature-dependent behaviour.

In Chapter 5 we explored two new Sn-based compounds (OBESnI_4 and EDBESnI_4),

which differed by the length of the organic cation. The change in length - hereby losing the ability to form hydrogen bonds between cations - resulted in large differences in crystal structure with a planar $\langle 100 \rangle$ -oriented perovskite for OBESnI_4 and a corrugated $\langle 110 \rangle$ -oriented perovskite for EDBESnI_4 . The difference in crystal structure resulted in strikingly different photoluminescence features with a narrow emission for OBESnI_4 and broad emission bands for EDBESnI_4 . Interestingly, thin films made of OBESnI_4 were black or red depending on the processing temperature and were correlated with a new crystal phase.

Hot-carrier solar cells could theoretically reach efficiencies of up to 66%, much higher than the 33% for single-junction solar cells. A key requirement is an active material with long hot-carrier cooling times. In this regard, Sn-based perovskites are promising with reported cooling into the nanosecond timescale. However, it is unclear how the composition affects this cooling and what the origin is of the long cooling times. In Chapter 6 we optimised the thin film deposition of three Sn-based perovskites containing formamidinium, methylammonium, and caesium to study the impact of the A-site cation. We reported hot-carrier cooling up to several nanoseconds at high fluence for the hybrid perovskites, while the fully inorganic perovskites showed sub-nanosecond cooling. This resulted in a blueshift of the photoluminescence peak at high excitation fluences up to ≈ 40 meV for the hybrid compounds and ≈ 10 meV for CsSnI_3 .

In Chapter 7 we continued the investigation to unravel the origin of these differences, where filling of the electronic band edges and a hot-phonon bottleneck have been proposed in the scientific literature to explain its origin. To distinguish between the mechanisms, we performed low-temperature photoluminescence and impulsive vibrational spectroscopy in conjunction with density functional theory and molecular dynamics calculations. The blueshift increases to > 100 meV at 4.4 K and is of similar magnitude in formamidinium- and caesium-based perovskites. The blueshift can be accounted for by band edge filling due to the low density of states near the band edge of Sn-based perovskites - setting Pb-based perovskites quite far apart. While the A-site cation plays a less fundamental role in hot-carrier cooling, it largely affects the vibrational dynamics. The coherent phonons in FASnI_3 dephase much faster compared to CsSnI_3 , implying a stronger phonon-phonon and electron-phonon scattering in the hybrid compound. Molecular dynamics support this notion with longer relaxation times in the fully inorganic system. With these works, we unravel the large differences in hot-carrier cooling times between Sn and Pb-based perovskites and identify the impact of the A-site cation on its properties, thereby creating new design rules to compose novel hot-carrier materials.

Samenvatting

De opwarming van de aarde zet zowel private als publieke belanghebbenden onder druk om meer hernieuwbare energiebronnen te gebruiken. Fotovoltaïsche (PV) zonnepaneeltechnologie, gekenmerkt door haar wijdverspreide beschikbaarheid en kosteneffectieve productie, zal de komende jaren nog belangrijker worden. De huidige PV-technologie op basis van silicium heeft echter beperkingen in zijn toepassingen. Daarentegen biedt de opkomende PV-technologie mogelijkheden voor toepassing in gebouwen en op daken van magazijnen, en voor het overtreffen van de efficiëntie van PV-panels die uitsluitend op silicium zijn gebaseerd.

Metaalhalogenide perovskieten staan in het middelpunt van de belangstelling van nieuwe PV-technologie, met een indrukwekkende toename in de efficiëntie van zonnecellen tot meer dan 25% in de afgelopen tien jaar. Er is echter een toenemende bezorgdheid over de stabiliteit en toxiciteit van de meest gebruikte op lood gebaseerde perovskieten. Strategieën om deze bezorgdheid weg te nemen zijn het toepassen van grote organische kationen voor een betere stabiliteit en structurele flexibiliteit, naast de vervanging van lood door het milieuvriendelijkere tin. Hoewel deze materialen succesvolle toepassingen hebben gevonden in zonnecellen en leds, staan veel van hun fundamentele eigenschappen ter discussie of zijn ze onbekend. Dit proefschrift behandelt de fotofysica van een uitgebreide reeks verbindingen, ontrafelt hun eigenschappen en draagt bij aan een beter begrip dat het rationele ontwerp van nieuwe materialen vergemakkelijkt.

In hoofdstuk 3 onderzochten we $(\text{PEA})_2\text{PbI}_4$ en $(3\text{-FPEA})_2\text{PbI}_4$ eenkristallen die zowel een smalle emissie in het groen als een brede emissie in het rood vertoonden. De brede emissie is onlangs in een aantal perovskiet-gerelateerde verbindingen waargenomen en de oorsprong ervan werd meestal aan zelfgevangen excitonen toegeschreven. In plaats daarvan toonden we aan dat de brede emissie afkomstig is van defecten in de bandkloof, waarmee we een veelvoorkomende misvatting in de wetenschappelijke gemeenschap uit de wereld helpen. Een cruciaal, maar eenvoudig, excitatie-experiment onder de bandkloof stelde ons in staat dit onderscheid te maken.

We vervolgden ons onderzoek naar $(\text{PEA})_2\text{PbI}_4$ en $(3\text{-FPEA})_2\text{PbI}_4$ in hoofdstuk 4 door de smalle emissieband te bestuderen met een hoge-resolutie en temperatuurafhankelijke fotoluminescentiestudie. De emissie aan de hoogenergetische kant van deze verbindingen wordt grotendeels beïnvloed door de keuze van het organische kation, waarbij de separatie van de hete-excitonresonanties wordt verkleind van ongeveer 40 meV tot ongeveer 15 meV voor respectievelijk $(\text{PEA})_2\text{PbI}_4$ en $(3\text{-FPEA})_2\text{PbI}_4$. Daarentegen is de emissie

aan de laagenergetische kant van de vrije excitonemissie afkomstig van excitonen die gebonden zijn aan defecten, zoals blijkt uit hun tijdsgeresolveerde verval en specifieke temperatuurafhankelijke gedrag.

In hoofdstuk 5 onderzochten we twee nieuwe op tin gebaseerde verbindingen (OBE-SnI₄ en EDBESnI₄), die verschilden door de lengte van het organische kation. De verandering in lengte - waardoor het vermogen om waterstofbruggen tussen kationen te vormen verloren gaat - resulteerde in grote verschillen in kristalstructuur met een vlakke $\langle 100 \rangle$ -georiënteerde perovskiet voor OBESnI₄ en een gegolfde $\langle 110 \rangle$ -georiënteerde perovskiet voor EDBESnI₄. Het verschil in kristalstructuur resulteerde in opvallend verschillende fotoluminescentiekenmerken met een smalle emissie voor OBESnI₄ en brede emissiebanden voor EDBESnI₄. Interessant is dat, afhankelijk van de verwerkingstemperatuur, dunne lagen gemaakt van OBESnI₄ zwart of rood waren en gecorreleerd werden met een nieuwe kristalfase.

Zonnecellen op basis van hete ladingdragers zouden theoretisch gezien een efficiëntie tot 66% kunnen bereiken, veel hoger dan de 33% voor zonnecellen met een enkelvoudige junctie. Een belangrijke vereiste is een materiaal met een lange koeltijd voor de hete drager. In dit opzicht zijn de op tin gebaseerde perovskieten veelbelovend met gerapporteerde koeltijden tot in de nanoseconde. Het is echter onduidelijk hoe de samenstelling deze koeling beïnvloedt en wat de oorzaak is van de lange koeltijden. In hoofdstuk 6 hebben we de dunne-laag depositie van drie op tin gebaseerde perovskieten met formamidinium, methylammonium en cesium geoptimaliseerd om de invloed van het A-kation te bestuderen. We rapporteerden koeling van de hete dragers tot enkele nanoseconden bij hoge fluentie voor de hybride perovskieten, terwijl de volledig anorganische perovskieten subnanoseconde-koeling vertoonden. Dit resulteerde in een blauwverschuiving van de fotoluminescentiepiek bij hoge excitatiefluentie tot ongeveer 40 meV voor de hybride verbindingen en ongeveer 10 meV voor CsSnI₃.

In hoofdstuk 7 hebben we het onderzoek voortgezet om de oorsprong van deze verschillen te ontrafelen, waarbij het opvullen van de elektronische bandranden en een hete-fonon knelpunt in de wetenschappelijke literatuur als verklaring zijn voorgesteld. Om onderscheid te maken tussen de mechanismen hebben we fotoluminescentie en impulsieve vibratiespectroscopie bij lage temperatuur uitgevoerd in combinatie met dichtheidsfunctionaaltheorie en moleculaire dynamica berekeningen. De blauwverschuiving neemt toe tot > 100 meV bij 4,4 K en is van vergelijkbare grootte in de op formamidinium en cesium gebaseerde perovskieten. De blauwverschuiving kan worden verklaard door vulling van de bandrand als gevolg van de lage dichtheid van staten van de op tin gebaseerde perovskieten - waardoor ze veel verschillen ten opzichte van de op lood gebaseerde perovskieten. Hoewel het kation op de A-locatie een minder fundamentele rol speelt in de koeling van de hete drager, beïnvloedt het in grote mate de trillingsdynamica. De coherente fononen in FASnI₃ defaseren veel sneller in vergelijking met CsSnI₃, wat een sterkere fonon-fonon en elektron-fonon verstrooiing in de hybride verbinding impliceert. Moleculaire dynamica ondersteunt dit idee met langere relaxatietijden in het volledig anorganische systeem. Met dit werk ontrafelen we de grote verschillen in hete-ladingdrager koeltijden tussen de op tin en de op lood gebaseerde perovskieten en identificeren we de invloed van het A-kation op de eigenschappen, waardoor nieuwe regels ontstaan voor het ontwerpen en samenstellen van nieuwe hete-ladingdrager materialen.

Curriculum Vitae

Education

- 2021-2023 University Teaching Qualification
University of Groningen, The Netherlands
- 2017-2019 Top Master Programme in Nanoscience (*cum laude*)
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- 2012-2016 Bachelor of Applied Physics (*cum laude*)
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Honours & Awards

- 2022 Best Oral Talk Award
Vlieland Conference
- 2017 - 2019 Top Master Programme in Nanoscience Scholarship, Zernike Institute for
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Conferences

- 2023 NanoGE HOPV, London, England, "Hot-Carrier Cooling in Sn-based Perovskites: State Filling and Hot-Phonon Bottlenecks", *Contributed talk*
- 2023 NWO Physics, Veldhoven, The Netherlands, "Compositional Engineering of Sn-based Perovskites for Long-Lived Hot-Carrier Materials", *Poster*
- 2023 NanoGE MATSUS Spring Meeting, Valencia, Spain, "Impact of Two Diammonium Cations on the Structure and Photophysics of Layered Sn-based Perovskites", *Contributed talk*
- 2022 Next-Gen PV V+, Groningen, The Netherlands, "Low-Energy Photoluminescence in Layered Tin Halide Perovskites: Unravelling the Impact of Diammonium Cations on the Structure and Its Photophysics.", *Contributed talk*
- 2022 Vlieland Conference, Vlieland, The Netherlands, "Compositional Engineering of Sn-based Perovskites for Hot-Carrier Solar Cells", *Contributed talk*
- 2022 MRS Spring Meeting, Honolulu, USA, "Impact of Diammonium Cations on the Structure and Photophysics of Sn-based Perovskites", *Contributed talk*
- 2022 MRS Spring Meeting, Honolulu, USA, "Understanding the Photophysics of Layered Lead Halide Perovskites", *Contributed talk*
- 2022 Physics@Veldhoven, Online event, "Understanding the photophysics of layered metal halide perovskites: extrinsic nature of broad and narrow emission bands", *Contributed talk*
- 2021 NanoGE Spring Meeting, Online event, "The impact of cation fluorination on the structure and photophysics of layered perovskites.", *Contributed talk*
- 2021 Physics@Veldhoven, Online event, "The impact of cation fluorination on the structure and photophysics of layered perovskites.", *Poster*
- 2020 NanoGE Fall Meeting, Online event, *Attendance*
- 2020 NanoGE TinPero, Online event, *Attendance*
- 2020 NanoGE HOPV, Online event, *Attendance*
- 2020 Physics@Veldhoven, Veldhoven, The Netherlands, "Ruddlesden-Popper perovskites revealing broad luminescence from sub-band gap states", *Poster*
- 2019 PSCO, Lausanne, Switzerland, "Ruddlesden-Popper perovskites revealing broad luminescence from sub-band gap states", *Poster*
- 2019 Next-Gen PV IV, Groningen, The Netherlands, "Ruddlesden-Popper perovskites revealing broad luminescence from sub-band gap states", *Poster*

List of Publications

1. **E.K. Tekelenburg**, F. Valduga de Almeida Camargo, A. Filippetti, A. Mattoni, L.J.M. Van de Ven, M. Pitaro, G. Cerullo and M.A. Loi, *Submitted*
2. L.J.M Van de Ven, **E.K. Tekelenburg**, M. Pitaro, J. Pinna, and M.A. Loi, *Submitted*
3. S. Mutalik, M. Ahmadi, J. Hong, **E.K. Tekelenburg**, P.P. Pescarmona, M.A. Loi, B.J. Kooi, L. Piveteau, L. Protesescu, *Submitted*
4. A.C Marx, H. Jafari, **E.K. Tekelenburg**, M.A. Loi, J. Slawinska, M.H.D. Guimarães, *Submitted*
5. W. Jin, C.-Y. Yang, R. Pau, Q. Wang, **E.K. Tekelenburg**, H.-Y. Wu, T. Liu, Q. He, Q. Li, R. Kroon, M.J. Heene, H.Y. Woo, M. Fahlman, M.A. Loi, and S. Fabiano, *In preparation*
6. L. Chen, **E.K. Tekelenburg**, K. Gahlot, M. Pitaro, J. Xi, A. Lasorsa, G. Feraco, L. Protesescu, P. Van der Wel, G. Portale, P. Rudolf, C.J. Brabec, and M.A. Loi, "In-situ SnSe deposition as passivation for scalable and stable quasi-2D lead-tin perovskite solar cells", *Energy and Environmental Science*, **2023**, Advanced Article
7. L. Chen, J. Xi, **E.K. Tekelenburg**, K. Tran, G. Portale, C.J. Brabec, and M.A. Loi, "Quasi-2D lead-tin perovskite memory devices based on blade coating", *Small Methods*, **2023**, 2300040
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* These authors contributed equally
12. H.-H. Fang, **E.K. Tekelenburg**, H. Xue, S. Kahmann, L. Chen, S. Adjokatse, G. Brocks, S. Tao, and M.A. Loi, "Unraveling the Broadband Emission in Mixed Tin-Lead Layered Perovskites", *Advanced Optical Materials*, **2022**, 2202038
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I have always been keen on having a good work-life balance, and for me, this means having nice activities with friends and family. The list below has become long but it simply underlines how important many people were outside of work in order for me to achieve the results I wanted at work.

To recuperate from a week in the lab, there is nothing better than evenings and weekends packed with board games. Justin, even though the rest of our friends have left Groningen, we continued to meet weekly on Wednesdays having dinner, discussions, and playing board games. It was nice to have such a good friend around to share the highs and lows of life.

Anton, we knew each other already for some years but started hanging out intensively during the pandemic. Who could have guessed that we would get close to obsessive behaviour with Pokémon TCG? I was always looking forward to these evenings weeks in advance - especially with a new deck - as a 7-year-old kid looking forward to his birthday. These evenings were the highlight of the week and gave me the energy to keep working productively at home or in the lab. You are an excellent friend. Joey, Max, Tim, Hugo and Jos, I am glad you were also as enthusiastic about Pokémon TCG as I was, and I think fondly back to the evenings we played over the years. Perhaps next time it will be in Utrecht instead of Groningen.

Floris and Loes I am glad to call you one of the best friends that I have. You are loyal, caring, and always enthusiastic. I am looking forward to meeting you more often when we are living in the same city.

Friederike, you have always been supportive and excited about me pursuing my PhD. It was probably your compliments and gentle nudges towards a PhD position that were decisive. It was always reassuring that you had confidence in me finishing my PhD.

Jasper, we have known each other since high school and have been friends since then. I appreciate your loyalty and support as well as the jokes you make. Your personality always makes me energised and makes it fun to be with.

James, Bas, Jelle, Sven, and Lennart you have known me for a long time and inviting me to DnD was one of the best choices you have made. Our WhatsApp group "Ultieme Nurd Eredivisie" nicely illustrates what we all love and I hope we will continue playing in the future. Perhaps during our next weekend trip with PJ, Olmo, and Ralph?

Ruud, Hidde, and Sjors, we started already during our student life playing Catan and have continued meeting in recent years. These days are always exciting with a lot of laughing, full of nonsense, and nothing short of worldly politics and intrigue (including trade bans with North Korea). Let's play another time soon!

The second-best thing to regain energy is playing sports like volleyball and road cycling. Without it, I would soon get moody and restless. I was lucky to have nice volleyball teammates over the years. They are too many to name them all but I would like to make a special mention of Anton, who convinced me to buy a racing bike, and my teammates Niels, Jasper, Yde, and Marvin who made my transition from Donitas to ON so much nicer. Leaving Groningen sadly also means leaving you behind.

Sometimes you need some support to find the motivation for doing some physical ex-

ercise. A good motivator to keep training is the yearly Satyr biking trip to the Ardennes with Diederick, Paul, Reinier, Sebastian, Pelle, Koen, Guus, and Olaf. Your company and competitiveness always ensure a good trip, not to forget the other memorable Satyr activities.

Niels, Jurre, and Tim thanks for the dinners, drinks and stories about PhD life at the Faculty of Medical Sciences. It was a relief to hear that my PhD life wasn't too bad compared to yours.

I also thank my former roommates at De Vlijt, friends from GHD Babylon, and my fellow KoCie members for the nice social gatherings and drinks.

My dear family, Tonnie, Monique, Xander and Lotte, Daniël and Roos, thank you for your continued support. You were never shy of giving good advice, helped me to keep my priorities straight and shared the joys of success. I can't wish for a nicer family, including new arrivals Fiene, Seppe, and Lima!

Sofie, you are the sweetest and I am so glad that we are very happy with each other after all these years, even when you had to endure all the nerdy (science) talk and me explaining physics-related topics. It was nice to see your genuine excitement about lasers when you began to work as an ophthalmologist and our work fields started to overlap, meaning that you could teach me a thing or two. You mean the world to me, and I am looking forward to the new chapter of our lives in Utrecht.

Eelco Kinsa Tekelenburg
Groningen
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