Multifunctionality of Layered Materials

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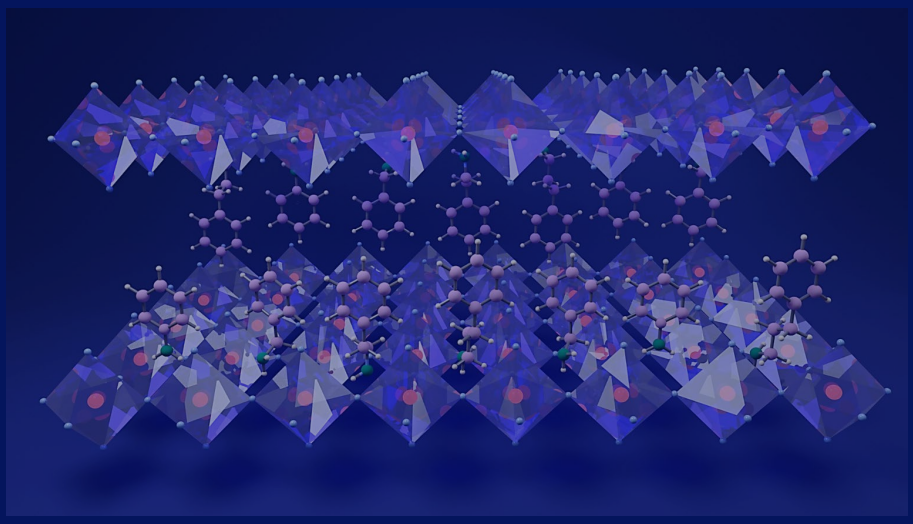
Appendices



Summary

Layered materials have attracted a lot of attention due to their multifunctionality. While many previous studies have focused on their optoelectronic properties, here we concentrate on the unique thermal, electrical and magnetic properties that arise from their two-dimensional connectivity. We use the term "layered materials" to describe compounds with relatively strong chemical bonding in two dimensions (in-plane) compared to much weaker interactions in the out-of-plane direction due to interlayer distances that are much larger than typical interatomic spacings. We introduce the layered materials studied in this thesis, as well as various physical properties related to these materials, in **Chapter 1**. We divide the compounds into two categories, comprised either of only inorganic elements, or a combination of organic and inorganic components forming hybrid compounds.

The first category includes the family of all-inorganic IV-VI semiconductors. These compounds are known to possess excellent thermoelectric properties, mainly for Pb-based compounds. A brief summary of thermoelectric properties and the many factors that affect their performance is given in **Chapter 1**. In **Chapter 3**, we investigate the emerging family of Ge-chalcogenides with their rich polymorphism. GeSe is a layered material with ultra-low thermal conductivity and thus shows potential for good thermoelectric performance. However, in practice the thermoelectric properties are unsatisfactory due to low electrical conductivity. We show that a rich polymorphism can be obtained by alloying GeSe with $AgBiTe_2$. Introducing $AgBiTe_2$ in the GeSe matrix induces the formation of a higher symmetry rhombohedral/cubic structure compared to the low symmetry orthorhombic structure of pristine GeSe. The change in crystal structure influences the electronic band structure, which affects electrical transport properties significantly and increases the thermoelectric figure of merit. Moreover, we find that some samples are metastable at low alloying concentration, for which the structure and thermoelectric properties change significantly after thermal treatment.

The second category of layered compounds studied here comprises tw0-dimensional organic-inorganic perovskites. We focus on their rich structural and magnetic properties. The

structures of layered hybrid perovskites are flexible and compatible with a wide range of both inorganic and organic components, which together dictate the physical properties that they possess and lead to a large variety of different structures and properties. **Chapter 4** focuses on structural analysis, where we investigate possible mechanisms by which inversion symmetry can be broken in layered organic-inorganic perovskites, especially those that contain non-Jahn-Teller (JT) active ions. We show that achieving a polar structure is not related to the buckling of MX_6 octahedra as thought to be the case for JT-active cations (such as Cu^{2+}), but is induced by the rotational degree of freedom of the organic cations, which influence the overall structure. We show that solving the crystal structures of these materials using X-ray diffraction data can be complicated by crystal twinning, which leads to difficulties in assigning the correct space group. This is also the case for the compound studied in **Chapter 6**.

We pay particular attention to investigating the magnetic properties of layered organic-inorganic perovskites. The Mn^{2+}-based layered perovskites (**Chapter 4 & 6**) show two-dimensional antiferromagnetism, which is manifested by a broad maximum in the magnetic susceptibility curve. At lower temperature they undergo long-range antiferromagnetic ordering; a weak ferromagnetic moment is exhibited due to spin canting. We demonstrate how varying the organic cation (**Chapter 4**), and halide concentration during synthesis (**Chapter 6**) affect the magnetic exchange energy. This quantity can be obtained by fitting the magnetic susceptibility versus temperature data to a 2D Heisenberg model for a square lattice. While varying the organic cation does not have any significant effect on the magnetic exchange energy, as shown in **Chapter 4**, changing the halide concentration during synthesis leads to a decreasing trend in exchange energy with increasing halide deficiency (**Chapter 6**). A possible explanation is due to local anion vacancies in the structure.

We demonstrate the presence of a large magnetocaloric effect in a Cu^{2+}-based layered organic-inorganic perovskite in **Chapter 5**. Such perovskites containing Cu^{2+} show long-range ferromagnetic ordering below a temperature that strongly depends on the applied magnetic field, indicating a large magnetic entropy change. We also study the nature of the magnetic phase transition in this material using a critical isotherm approximation, which is consistent with the 2D-XY spin model.

In summary, this thesis shows that layered materials possess many functionalities for which the structure and physical properties are highly correlated. By studying only a few selected layered materials, we demonstrate that this research area is vast, and that many unique properties can be found only in materials with two-dimensional chemical connectivity.

Samenvatting

Gelaagde materialen hebben door hun multifunctionaliteit veel aandacht ontvangen. Waar veel voorgaande studies zich focusten op de opto-elektronsiche eigenschappen, concentreren wij ons hier op de unieke thermische, elektrische en magnetische eigenschappen die voortkomen uit de tweedimensionale connectiviteit van de materialen. We gebruiken de term "gelaagde materialen" om verbindingen aan te geven die sterkere chemische bindingen in twee dimensies (in het vlak) vertonen dan de veel zwakkere interacties in de richting haaks op het vlak. Deze zwakke interacties worden veroorzaakt doordat de afstanden tussen de lagen veel groter zijn dan typische interatomische afstanden. We introduceren de gelaagde materialen, en verscheidene gerelateerde fysieke eigenschappen die bestudeerd worden, in **Hoofdstuk 1** van dit proefschrift. We verdelen de verbindingen in twee categorieën: verbindingen opgebouwd uit alleen anorganische elementen en hybride verbindingen opgebouwd uit een combinatie van organische en anorganische elementen.

De eerste categorie bevat de familie van volledig anorganische IV-VI halfgeleiders. Deze verbindingen, en voornamelijk op Pb gebaseerde materialen, staan bekend om hun excellente thermo-elektrische eigenschappen. Een beknopte samenvatting van de thermo-elektrische eigenschappen, en de vele factoren die de prestaties beïnvloeden, wordt gegeven in **Hoofdstuk 1**. In **Hoofdstuk 3** onderzoeken wij de opkomende familie van Ge-chalcogenides met hun rijke polymorfisme. GeSe is een gelaagd materiaal dat een zeer lage warmtegeleidingscoëfficiënt vertoont en daardoor de potentie heeft voor goede thermo-elektrische prestaties. Echter, in de praktijk zijn de thermo-elektrische prestaties teleurstellend door hun lage elektrische geleidbaarheid. Wij laten zien dat een rijke polymorfisme verkregen kan worden door een legering te maken van GeSe met $AgBiTe_2$. De introductie van $AgBiTe_2$ in de GeSe matrix induceert de formatie van een rhombohedrale/cubische kristalstructuur met een hogere symmetrie dan de laag symmetrische orthorhombische kristalstructuur van zuiver GeSe. De verandering van de kristalstructuur beïnvloedt de elektronische bandstructuur en hierdoor ook mede de elektrische transport eigenschappen significant en verhoogt het thermo-elektrische prestatiegetal. Bovendien constateren wij dat verscheidene monsters metastabiel zijn bij lage legering concentraties, waarbij de structuur en thermo-elektrische eigenschappen veranderen na een thermische behandeling.

De tweede categorie van gelaagde materialen die hier bestudeerd worden bestaan uit tweedimensionale organisch-anorganische perovskieten. We concentreren ons op hun rijke structurele en magnetische eigenschappen. De structuren van gelaagde hybride perovskieten zijn flexibel en compatibel met een wijde selectie aan anorganische en organische componenten, die samen de fysieke eigenschappen dicteren en leiden tot een grote verscheidenheid aan structuren en eigenschappen. **Hoofdstuk 4** richt zich op structurele analyse, waar wij de mogelijke mechanismen onderzoeken waardoor de inversie symmetrie van gelaagde organisch-anorganisch perovskieten, die voornamelijk niet-Jahn-Teller (JT) actieve ionen bevatten, kan worden gebroken. Wij laten zien dat het stabiliseren van een polaire structuur niet gerelateerd is aan het knikken van de MX_6 octaëders, zoals in het geval van JT-actieve ionen (zoals Cu^{2+}) gedacht wordt, maar door de rotationele vrijheidsgraad van de organische kationen die de volledige structuur beïnvloeden. Wij laten zien dat het oplossen van de kristalstructuren door middel van eenkristalröntgendiffractiedata gecompliceerd kan worden door de aanwezigheid van kristallografische tweelingen, wat leidt tot moeilijkheden bij het toewijzen van de correcte ruimtegroep. Dit is ook het geval voor het materiaal wat bestudeerd wordt in **Hoofdstuk 6**.

Wij onderzoeken ook nadrukkelijk de magnetische eigenschappen van gelaagde organisch-anorganische perovskieten. De gelaagde perovskieten op basis van Mn^{2+} (**Hoofdstuk 4** en **6**) vertonen tweedimensionaal antiferromagnetisch gedrag, wat zich manifesteert als een breed maximum in de magnetische susceptibiliteit kromme. Bij lage temperaturen ordenen zij antiferromagnetisch. Een zwak ferromagnetisch moment wordt waargenomen als gevolg van het kantelen van de spin. Wij demonstreren hoe het variëren van het organisch kation (**Hoofdstuk 4**), en de halide concentratie gedurende de synthese (**Hoofdstuk 6**), de magnetische uitwisselingsenergie beïnvloedt. Deze kwantiteit kan worden verkregen door de magnetische susceptibiliteit versus de temperatuur kromme te fitten aan een 2D Heisenberg model voor een vierkant rooster. Hoewel het variëren van het organisch kation geen significant effect heeft op de magnetische uitwisselingsenergie, zoals aangetoond wordt in **Hoofdstuk 4**, leidt het veranderen van de halide concentratie tijdens de synthese (**Hoofdstuk 6**) tot een dalende trend in de magnetische uitwisselingsenergie met een groter wordende halide tekort. Een mogelijke verklaring hiervoor zijn lokale anionische leegtes in de structuur die hierdoor ontstaan.

Wij demonstreren de aanwezigheid van een groot magneto-calorisch effect in een gelaagde organisch-anorganisch perovskiet op basis van Cu^{2+} in **Hoofdstuk 5**. Dergelijke perovskieten, die Cu^{2+} bevatten, vertonen ferromagnetische ordening beneden een kritische temperatuur die sterk afhankelijk is van de toegepaste magnetisch veldsterkte. Dit is een indicatie voor een grote verandering van de magnetische entropie. Ook onderzoeken wij de aard van de magnetische transitie in dit materiaal met behulp van een kritische isotherm benadering die consistent is met het 2D-XY spin model.

Samenvattend, dit proefschrift laat zien dat gelaagde materialen veel functionaliteiten bevatten waarbij de structuur en fysieke eigenschappen in hoge mate gecorreleerd zijn. Door het bestuderen van alleen een kleine greep gelaagde materialen demonstreren wij dat dit onderzoeksveld enorm is en dat vele unieke eigenschappen enkel gevonden kunnen worden in materialen met tweedimensionale chemische connectiviteit.

List of Publications

Polar Structure and Two-Dimensional Heisenberg Antiferromagnetic Properties of Arylamine-based Manganese Chloride Layered Organic-Inorganic Perovskites
[Chapter 4]
L. Septiany, D. Tulip, M. Chislov, J. Baas, G.R.Blake. *Inorganic Chemistry.* **2021**. In Press.

Magnetocaloric Effect and Critical Behaviour in Arylamine-based Copper Chloride Layered Organic-Inorganic Perovskite.
[Chapter 5]
L. Septiany, G. R. Blake. *Journal of Magnetism and Magnetic Materials*. **2021**. In Press.

Thermoelectric Properties and Phase Evolution of $(GeSe)_{1-x}(AgBiTe_2)_x$ Alloys.
[Chapter 3]
L. Septiany, J. Baas, G.R. Blake. *In Preparation*

A Novel Antiferromagnet Spinel: $MnRh_2O_4$.

L. Septiany, A. Syariati, J. Baas, P. Rudolf, G.R. Blake. *In Preparation*

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Liany Septiany

"If you can dream it, you can do it." – Walt Disney