

**FINAL REPORT****1 General information****DFG reference number:** KI 2865/1-1**Project number:** 529795573**Project title:** *Physikalische und technologische Grundlagen zur Gewinnung von ZnS:Mn Nanopulver, Metallopolymeren und Heterostrukturen auf Basis von nanoporösem Silizium***Name of the applicant:** Professor Dr. Valerii Kidalov**Official address(es):**

Technische Universität Dortmund

Fakultät Physik

Lehrstuhl für Experimentelle Physik II

Otto-Hahn-Straße 4

44227 Dortmund

**Name(s) of the host individual and institution:**

Prof. Dr. Marc Aßmann

Technische Universität Dortmund

Fakultät Physik, Experimentelle Physik II

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## 2 Summary

Silicon has served as the cornerstone of modern electronics for decades due to its low cost and mature processing technologies. However, its physical limitations – in particular its inability to operate reliably at high temperatures, under intense radiation and in demanding optoelectronic regimes – necessitate the exploration of wide-bandgap semiconductors. Silicon carbide (SiC), gallium oxide ( $\beta$ -Ga<sub>2</sub>O<sub>3</sub>) and zinc oxide (ZnO) are among the most promising candidates for next-generation high-performance device. A central challenge is their integration with silicon platforms. Direct deposition of SiC or  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> on crystalline Si leads to severe lattice and thermal-expansion mismatches, high defect densities and degraded electronic properties. In this context, our study introduced porous silicon as a compliant buffer and examined the additional effect of an intermediate SiC layer. Using RF magnetron sputtering, we fabricated and analysed four heterostructures: Ga<sub>2</sub>O<sub>3</sub>/Si, Ga<sub>2</sub>O<sub>3</sub>/por-Si/Si, Ga<sub>2</sub>O<sub>3</sub>/SiC/por-Si/Si(100) and Ga<sub>2</sub>O<sub>3</sub>/SiC/por-Si/Si(111). Scanning electron microscopy, X-ray diffraction, Raman spectroscopy and finite-element simulations show that Ga<sub>2</sub>O<sub>3</sub>/SiC/por-Si/Si(100) provides the most favourable structural characteristics, with reduced defect formation, improved crystalline order and the strongest Raman response, confirming the effectiveness of the SiC/por-Si dual buffer in stress relaxation. These results highlight the potential of engineered buffer systems for reliable, cost-effective integration of wide-bandgap oxides with silicon technology in power electronics, renewable energy and radiation-resistant devices.

Within the project, luminescent Nd<sup>3+</sup>  $\beta$ -diketonate complexes were investigated by optical spectroscopy in combination with theoretical analysis, including Judd–Ofelt/Sinha treatment of the f–f transitions. The data indicate predominantly eight-coordinate Nd<sup>3+</sup> environments with distorted square-antiprismatic geometries and a non-cubic ligand field, as evidenced by characteristic band splitting and transition intensities. An increase in Nd–O bond covalence correlates with enhanced near-infrared emission. Complexes containing the unsaturated dmhpd ligand show superior luminescent performance, whereas bulky aromatic substituents tend to suppress emission by shielding the lanthanide ion. The main conclusion is that rational variation of  $\beta$ -diketonate ligands provides an effective handle to tune the NIR photophysical properties of Nd<sup>3+</sup> complexes, making them promising components for optical fibres and precursors for scintillating materials.

Silizium hat aufgrund seiner niedrigen Kosten und der ausgereiften Prozesstechnologien seit Jahrzehnten als Eckpfeiler der modernen Elektronik gedient. Seine physikalischen Einschränkungen – insbesondere die mangelnde Zuverlässigkeit bei hohen Temperaturen, unter intensiver Strahlung und in anspruchsvollen optoelektronischen Betriebsregimen – machen jedoch die Erforschung von Halbleitern mit großer Bandlücke notwendig. Siliziumkarbid (SiC), Galliumoxid ( $\beta$ -Ga<sub>2</sub>O<sub>3</sub>) und Zinkoxid (ZnO) zählen zu den vielversprechendsten Kandidaten für Hochleistungsbauelemente der nächsten Generation. Eine zentrale Herausforderung besteht in ihrer Integration in Siliziumplattformen. Die direkte Abscheidung von SiC oder  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> auf kristallinem Silizium führt zu starken Gitter- und thermischen Ausdehnungsfehlanspassungen, zu hohen Defektdichten und zu verschlechterten elektronischen Eigenschaften. Vor diesem Hintergrund führte unsere Studie poröses Silizium als nachgiebige Pufferschicht ein und untersuchte zusätzlich den Effekt einer zwischengeschalteten SiC-Schicht. Unter Verwendung von HF-Magnetronspütern wurden vier Heterostrukturen hergestellt und analysiert: Ga<sub>2</sub>O<sub>3</sub>/Si, Ga<sub>2</sub>O<sub>3</sub>/por-Si/Si, Ga<sub>2</sub>O<sub>3</sub>/SiC/por-Si/Si(100) und Ga<sub>2</sub>O<sub>3</sub>/SiC/por-Si/Si(111). Untersuchungen mittels Rasterelektronenmikroskopie, Röntgendiffraktometrie, Raman-Spektroskopie und Finite-Elemente-Simulationen zeigen, dass Ga<sub>2</sub>O<sub>3</sub>/SiC/por-Si/Si(100) die günstigsten strukturellen Eigenschaften aufweist, mit verringerter Defektbildung, verbessertem kristallinem Ordnungsgrad und der stärksten Raman-Antwort, was die Effektivität der SiC/por-Si-Doppelpufferschicht bei der Spannungsrelaxation bestätigt. Diese Ergebnisse unterstreichen das Potenzial gezielt entwickelter Puffersysteme für eine zuverlässige und kosteneffiziente Integration von Wide-Bandgap-Oxiden in die Siliziumtechnologie für Leistungselektronik, erneuerbare Energien und strahlungsresistente Bauelemente.

Im Rahmen des Projekts wurden lumineszierende  $\beta$ -Diketonatkomplexe des Nd<sup>3+</sup> mittels optischer Spektroskopie in Kombination mit theoretischer Analyse untersucht, einschließlich einer Auswertung der f-f-Übergänge nach dem Judd-Ofelt/Sinha-Ansatz. Die Daten deuten auf überwiegend achtfach koordinierte Nd<sup>3+</sup>-Umgebungen mit verzerrter quadratisch-antiprismatischer Geometrie und einem nicht-kubischen Ligandenfeld hin, was sich im charakteristischen Bandenaufspalten und in den Übergangsintensitäten widerspiegelt. Eine Zunahme der Kovalenz der Nd-O-Bindung korreliert mit einer verstärkten Emission im nahen Infrarot. Komplexe mit dem ungesättigten dmhpd-Liganden zeigen die besten Lumineszenzeigenschaften, wohingegen voluminöse aromatische Substituenten zur Unterdrückung der Emission neigen, da sie das Lanthanoidion abschirmen. Die Hauptschlussfolgerung besteht darin, dass die gezielte Variation der  $\beta$ -Diketonatliganden ein wirksames Steuerinstrument zur Einstellung der NIR-photophysikalischen Eigenschaften von Nd<sup>3+</sup>-Komplexen darstellt und sie zu

vielversprechenden Komponenten für optische Fasern und Vorläufermaterialien für Szintillatoren macht.

### 3 Scientific Progress Report

Wide-bandgap semiconductors (GaN, AlN, ZnO) and the ultra-wide-bandgap oxide  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> are key materials for light sources, sensors and power electronics, but their potential is limited by the lack of high-quality epitaxial films on inexpensive substrates. The main bottlenecks are strong lattice and thermal-expansion mismatch and the high cost of suitable crystalline substrates. Our previous work has shown that porous silicon and SiC films obtained by atomic substitution can substantially reduce mechanical stresses and improve the quality of ZnO and  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> films on silicon. In parallel, ZnS:Mn and ZnS:Cu powders obtained by self-propagating high-temperature synthesis (SHS) have been studied to understand how particle size affects spectral-luminescent properties. A further line of research concerns coordination compounds and metallopolymers based on Nd<sup>3+</sup> and Gd<sup>3+</sup>  $\beta$ -diketonates, which combine high thermal stability with efficient luminescence and are promising for emissive layers in organic electroluminescent devices and for magneto-optical applications.

#### Aims of the project

The project combines three closely related directions:

(1) To optimise the fabrication of SiC films with defined thickness on porous silicon and to create SiC/por-Si/Si, ZnO/SiC/por-Si/Si and  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>/SiC/por-Si/Si heterostructures, followed by a comprehensive study of their structural, electrical and optical properties. The goal is to obtain low-stress SiC buffer layers that act as universal templates for wide-bandgap materials.

(2) To obtain micro-, meso- and nanopowders of ZnS:Mn and ZnS:Cu by SHS, to investigate their structural and optical characteristics, and to study the influence of magnetic field on the photoluminescence of centres of different nature under confined geometry (small particles with a large fraction of surface states).

(3) To synthesise new multifunctional metallopolymers based on Nd(III) and Gd(III)  $\beta$ -diketonate complexes, to study their structure, luminescent and magneto-optical properties, and to clarify how the crystal field and magnetic field affect intraconfigurational f–f transitions.

Taken together, the project aims to establish the scientific and technological foundations for wide-bandgap heterostructures on silicon and for functional luminescent materials for optoelectronics and sensing

**Published results:**

Below we briefly summarise our project-related publications [1–7], emphasising the main novelties and their internal coherence.

Publication [1] shows for the first time that mechanical stresses in SiC films grown on porous Si are strongly reduced compared with SiC on monocrystalline Si and clarifies the mechanism of SiC formation. Quantitative agreement between XRD/Raman stresses and thermoelastic finite element method (**FEM**) establishes SiC/porous-Si heterostructures as suitable substrates for epitaxial wide-bandgap semiconductors.

Publication [2] uses tip-enhanced Raman spectroscopy (**TERS**) combined with AFM, near-field IR mapping and conventional Raman to directly correlate local morphology with local vibrational and optical responses in SiC/porous-Si/Si.

For  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>, publications [3–5] demonstrate the advantages of compliant underlayers based on porous Si and SiC/porous-Si/Si compared with “rigid” Si. Systematic comparison of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> on Si, porous-Si/Si and three-layer SiC/porous-Si/Si identifies the latter as providing the best structural quality and most homogeneous stress state, while a Raman spectral-decomposition procedure enables quantitative stress determination in multilayer stacks.

Publication [6] extends the stress-engineered template concept to ZnO, demonstrating textured growth, reduced defect density and improved structural parameters on SiC/porous-Si/Si and confirming the universal character of this platform.

Our conference contribution [7] addresses luminescent ZnS:Mn powders, where we investigate magnetic-field effects on emission and demonstrate magnetic-field control of the luminescent response.

Together, publications [1–6] form a logical sequence from SiC/porous-Si/Si templates to  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> and ZnO films grown on these templates.

**Unpublished results:**

In our unpublished works [15,19], spin-dependent magnetoluminescence is investigated in highly dispersed ZnS powders doped with Mn and Cu at cryogenic temperatures, while a unified synthetic and spectroscopic platform is established for NIR-emissive Nd(III)  $\beta$ -diketonate metallopolymers with controlled coordination environment and morphology.

Highly dispersed ZnS:Mn and ZnS:Cu phosphors are classical II–VI semiconductor systems in which Mn<sup>2+</sup> ions give rise to a broad orange  ${}^4T_1 \rightarrow {}^6A_1$  emission band, while Cu-related centres are responsible for blue–green bands associated with intrinsic defects, donor–acceptor complexes and three-centre Auger-type processes [8]. Their luminescent properties

are known to depend sensitively on dopant concentration, synthesis conditions and defect structure, which control the balance between host-related and impurity-related recombination channels [9–12]. These materials are widely used in powder and thin-film electroluminescent devices, where the interplay between radiative centres, killer impurities and charge transport has been extensively analysed [11,12]. Against this background, magnetic-field effects on luminescence in doped ZnS have attracted considerable interest as a sensitive probe of spin-dependent energy transfer and recombination involving Mn-related 3d states and defect complexes in wide-bandgap II–VI semiconductors [10,13,14].

In our unpublished work [15], magnetic-field effects on the luminescence of highly dispersed Mn- and Cu-doped ZnS powders synthesised by self-propagating high-temperature synthesis were investigated by low-temperature photoluminescence at  $T = 1.6$  K in magnetic fields up to 5 T. For ZnS:Mn, the PL spectra are dominated by the broad orange  ${}^4T_1 \rightarrow {}^6A_1$  emission band of  $Mn^{2+}$  ions near 580–590 nm; increasing the magnetic field leads to a pronounced change in its integrated intensity, whereas the peak position and spectral shape remain essentially unchanged. In ZnS:Cu powders a broad green band around 520–530 nm, attributed to  $Cu^+$  centres and Cu-related donor–acceptor complexes, exhibits strong negative magnetoluminescence: its integrated intensity decreases by more than a factor of two between 0 and 5 T, again without a detectable spectral shift. The absence of measurable changes in spectral position despite large intensity variations shows that the magnetic field primarily affects spin-dependent energy-transfer and carrier-capture processes (donor–acceptor pairs, traps) competing with radiative recombination at  $Mn^{2+}$  or  $Cu^+$  centres, rather than the band gap of ZnS or the intrinsic energetic structure of the emitting centres. The comparative behaviour of ZnS:Mn and ZnS:Cu thus demonstrates that magnetoluminescence in highly dispersed ZnS powders is extremely sensitive to the microscopic nature of the luminescent centres and their coupling to spin-dependent transfer and trapping processes [15].

The observed decrease in PL intensity in an external magnetic field can be explained by a slowdown of the energy transfer to  $Mn^{2+}$  centres caused by Zeeman splitting of the electronic levels [13,14]. The splitting leads to spin polarisation (“magnetic freezing”) of carriers and  $Mn^{2+}$  ions, so that the upper Zeeman components of the band states and of the 3d levels of  $Mn^{2+}$  no longer participate efficiently in spin-dependent sp–d energy transfer. At helium temperatures in II–VI compounds only the lowest electronic and hole sublevels are expected to be populated, and a magnetic field further splits the ground  ${}^6A_1$  and excited  ${}^4T_1$  terms of  $Mn^{2+}$  into 6 and 4 sublevels, respectively [13,14]. Energy transfer involving exciton annihilation and

excitation of the  $\text{Mn}^{2+}$  3d shell must obey conservation of the total angular momentum of the coupled exciton– $\text{Mn}^{2+}$  system; as electrons are gradually frozen into the lowest Zeeman sub-levels, spin configurations that allow efficient energy transfer become depopulated. This reduces the rate of sp–d energy transfer and manifests itself as a strong quenching of the  $\text{Mn}^{2+}$ -related luminescence intensity.

In our unpublished work [19], we investigate the effect of temperature and coordination environment on the luminescent properties of  $\beta$ -diketonate systems containing neodymium(III) and gadolinium(III) ions. In the present section, this approach is generalised by introducing a systematic series of Nd(III)  $\beta$ -diketonate systems based on the same unsaturated ligand dmhpd:  $\text{Nd}(\text{dmhpd})_3$ ,  $\text{Nd}(\text{dmhpd})_3\cdot\text{Phen}$ , and the corresponding metallopolymers  $[\text{Nd}(\text{dmhpd})_3]_n$  and  $[\text{Nd}(\text{dmhpd})_3\cdot\text{Phen}]_n$ , complemented by mphpd-based analogues. Here we focus on the Nd(III)-based complexes. A one-pot free-radical polymerisation in DMF at  $\approx 80^\circ\text{C}$  (AIBN) converts pre-formed mono- and heteroligand complexes into main-chain metallopolymers with a high density of coordination nodes. IR spectroscopy and thermal analysis demonstrate that in all systems the  $\beta$ -diketonate acts as a bidentate chelating ligand, the  $\text{Nd}^{3+}$  coordination number is eight, phenanthroline replaces coordinated water without altering the distorted square-antiprismatic geometry, and polymerisation does not significantly perturb the local coordination environment.

Diffuse-reflectance and absorption spectra were used to extract Sinha covalency parameters and Judd–Ofelt intensity parameters ( $\Omega_2$ ,  $\Omega_4$ ,  $\Omega_6$ ) for the hypersensitive  $^4I_{9/2} \rightarrow ^2G_{7/2}$ ,  $^4G_{5/2}$  transition. Variations in Nd–O covalency and coordination-sphere symmetry were correlated with the evolution of NIR emission along the  $\text{Nd}(\text{dmhpd})_3 \rightarrow \text{Nd}(\text{dmhpd})_3\cdot\text{Phen} \rightarrow [\text{Nd}(\text{dmhpd})_3]_n \rightarrow [\text{Nd}(\text{dmhpd})_3\cdot\text{Phen}]_n$  series and their mphpd analogues. Nd–O bond covalency and ligand-field polarisability increase when going from monoligand to heteroligand complexes and upon the introduction of phenanthroline; the highest covalency, strongest ligand-field effects and most intense NIR emission are observed for  $[\text{Nd}(\text{dmhpd})_3\cdot\text{Phen}]_n$ .

The luminescence of all mono- and polymeric complexes was investigated at 293 K and 1.6 K. All systems exhibit the characteristic  $\text{Nd}^{3+}$   $^4F_{3/2} \rightarrow ^4I_{9/2}$ ,  $^4I_{11/2}$  and  $^4I_{13/2}$  transitions ( $\approx 870$ – $900$ ,  $1050$ – $1070$ ,  $1320$ – $1350$  nm). Cooling to 1.6 K sharpens the spectra, resolves Stark components and reveals weak phonon replicas shifted by  $\approx 100$ – $120$   $\text{cm}^{-1}$ , indicative of pronounced electron–phonon coupling in the metal–organic environment. Heteroligand complexes  $\text{NdL}_3\cdot\text{Phen}$  and their metallopolymers generally exhibit higher emission intensity than their monoligand counterparts, consistent with the antenna role of phenanthroline and with the Judd–Ofelt analysis.

SEM analysis shows that morphology depends on both the ligand and the occurrence of polymerisation: monomeric complexes form nearly spherical particles ( $\leq 50$  nm), whereas dmhpd-based metallopolymers develop layered, partially porous morphologies with limited agglomeration. For  $[\text{Nd}(\text{dmhpd})_3]_n$  and  $[\text{Nd}(\text{dmhpd})_3 \cdot \text{Phen}]_n$ , the rigid layered frameworks correlate with modified emission behaviour, indicating that morphology and packing can either facilitate or hinder energy migration and quenching. Taken together, these results establish a unified synthetic and spectroscopic route to highly loaded Nd(III) metallopolymers with a controlled coordination environment, tunable covalency and tailored morphology for NIR photonics, optical amplifiers and polymer-based scintillators, significantly extending earlier proof-of-concept studies on partially loaded copolymers [16–18]. An analogous suite of structural and spectroscopic investigations was also carried out for the corresponding isostructural Gd(III) complexes, confirming their close structural similarity to the Nd(III) series and providing a robust basis for comparative analysis of coordination geometry, morphology and luminescence behaviour.

Together, our unpublished studies [15,19] demonstrate, first, that magnetoluminescence in highly dispersed Mn- and Cu-doped ZnS powders provides a sensitive probe of spin-dependent energy transfer and recombination in wide-bandgap II–VI semiconductors, and second, that Nd(III)  $\beta$ -diketonate metallopolymers offer a tunable platform where coordination environment, Nd–O covalency and morphology can be systematically linked to NIR emission efficiency. Taken together, these results extend the project beyond inorganic wide-bandgap films to magnetic-field-sensitive phosphors and lanthanide-based metallopolymers for NIR photonics.

### Deviations from the Original Concept

No deviations from the original concept were observed during the project implementation

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## 4 Published Project Results

### 4.1 Category A – Articles in peer-reviewed journals, contributions to peer-reviewed conferences or to anthology volumes, and book publications

[A1] **Kidalov, V. V.**; Revenko, A. S.; Duleba, D.; Redko, R. A.; Aßmann, M.; Gudymenko, O. I.; Johnson, R. P. Investigation of mechanical stresses in SiC/Porous-Si heterostructure. *ECS J. Solid State Sci. Technol.* 13(11), 114003 (2024). <https://doi.org/10.1149/2162-8777/ad89f8>

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### 4.2 Category B – Any other form of published results

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### **4.3 Patents (applied for and granted)**