Annual Report

Leibniz-Institut für Kristallzüchtung

im Forschungsverbund Berlin e.V.

2017

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2017

Preface

Liebe Leserinnen und Leser, Liebe Kolleginnen und Kollegen,

es ist mir eine große Freude, Sie als neuer wissenschaftlicher Leiter des Leibniz-Instituts für Kristallzüchtung an dieser Stelle begrüßen zu dürfen. Das Jahr 2017 erwies sich für das IKZ als ein Jahr des Umbruchs - nach fast vier Jahren der Übergangszeit empfahl die Berufungskommission der Humboldt-Universität zu Berlin im August 2017 meine Kandidatur zum wissenschaftlichen Direktor, die ich offiziell am 1. Februar 2018 angetreten habe.

An dieser Stelle möchte ich mich als erstes im Namen aller Mitarbeiterinnen und Mitarbeiter des IKZ bei Prof. Dr. Günther Tränkle für sein Engagement und die Arbeit am IKZ in all diesen Jahren bedanken. Es gelang ihm auf eindrucksvolle Weise, die Leitung des IKZ mit den anspruchsvollen Aufgaben als wissenschaftlicher Direktor am Ferdinand-Braun-Institut (FBH) zu verbinden.

Dank der Erfahrung und Belastbarkeit von Günther Tränkle blieb die F&E-Tätigkeit am IKZ auf einem hohen und international führenden Niveau. Die Jahre unter seiner Leitung haben die Verbindung zwischen dem IKZ und dem FBH gestärkt, die zweifellos eine gute Basis für zukünftige produktive Kooperationen bilden wird, die für beide Leibniz-Institute von gegenseitigem Nutzen sein werden.

Auf wissenschaftlicher und technologischer Ebene ist die Eröffnung des Zentrums für Lasermaterialien zweifellos ein Highlight des Jahres. Das Zentrum erschließt neue F&E-Möglichkeiten für das IKZ und stärkt die Kompetenzen des Instituts im Bereich der Kristalle für die Photonik. Bitte werfen Sie einen Blick auf den Highlight Abschnitt dieses, um mehr über die Geschichte hinter dem Zentrum und seine Zukunftspläne zu erfahren.

2017 haben wir unsere Aktivitäten auf dem Gebiet der Oxidmaterialien für neuartige Elektronik intensiviert, indem wir mehrere neue Projekte zur angewandten und grundlegenden Forschung an Galliumoxid sowie anderen Oxidverbindungen zur Verbreiterung der Materialbasis begonnen haben. Zur Unterstützung dieser Forschungsaktivitäten erhielt das IKZ 2017 erhebliche EFRE-Mittel für die Einrichtung eines „Applikationslabors für Materialien der Oxidelektronik“.

Mit seiner Expertise für das Wachstum hochreiner, isotopisch reiner Germaniumkristalle im Jahr 2017 schloss sich das IKZ dem internationalen LEGEND-Projekt an, das einen internationalen Rahmen für kollaborative Studien zum neutrinolosen Doppel-Beta-Zerfall von ^{76}Germanium bietet, einem Zerfallsprozess, der zur Lösung der Geheimnisse der Teilchenphysik beitragen könnte.


Photo: Tina Merkau © IKZ

Neben dem wissenschaftlichen Fortschritt gab es im Jahr 2017 viele Erfolge auf personeller Ebene: Das Spin-off-Unternehmen der IKZ, die GOLARES GmbH, erhielt den Gründerpreis der Leibniz-Gemeinschaft, Dr. Kaspars Dadzis erhielt den Nachwuchspreis der LIMTECH-Allianz und drei unserer Doktoranden verteidigten erfolgreich ihre Doktorarbeit.

All dies wäre ohne das Engagement und die Kompetenz unserer Mitarbeiterinnen und Mitarbeiter nicht möglich gewesen. Deshalb möchte ich mich bei allen Kolleginnen und Kollegen des IKZ bedanken, die diese Erfolgsgeschichten im Jahr 2017 ermöglicht haben! Ich möchte auch dem Land Berlin und der Bundesregierung für die finanzielle Unterstützung danken, die die Forschungsaktivitäten des Instituts ermöglicht.

Das Institut bereitet sich nun auf die im Dezember 2018 stattfindende Evaluierung vor, in die die Forschungs- und Entwicklungsfortschritte der letzten sieben Jahre einfließen. Im nächsten Jahr werden wir auch die ersten Schritte zur Umsetzung der neuen Institutsstrategie unternehmen. Ich freue mich auf die Zusammenarbeit mit unseren internen und externen Kolleginnen und Kollegen und Partnern bei diesen spannenden zukünftigen Herausforderungen!

Ich wünsche Ihnen eine unterhaltsame Lektüre

Mit freundlichen Grüßen

Thomas Schröder

Preface

Dear Readers and Colleagues,

It is a great privilege for me to address you as the new scientific director of the Leibniz Institut für Kristallzüchtung. The year 2017 turned out to be for the IKZ the year of change – after almost four years of the interim period, the appointment committee of the Humboldt Universität zu Berlin approved my candidature to become the scientific director in August 2017, which I started officially on 1st February, 2018.

Firstly, on behalf of all IKZ employees, I would like to thank Prof. Günther Tränkle for his commitment and the work at the IKZ during all these years. He succeeded to combine the activities for the IKZ with the demanding duties as the scientific director at the Ferdinand-Braun Institute (FBH).

Based on Prof. Tränkle´s experience and resilience, the R&D activity at the IKZ remained on a high and leading international level. The years under the lead of Günter Tränkle have strengthened the link between the IKZ and FBH, which – without doubt – will form the firm basis for future productive collaborations, bringing mutual benefits for both Leibniz institutes.

On a science and technology level, the launch of the Center for Laser Materials without doubts became the highlight of the year, attracting to the IKZ new R&D opportunities and strengthening its expertise on the technology of crystals for photonics. Please, take a look in the Highlight section of this report to learn the story behind the Center and its future plans.

In 2017, we intensified our activities in the field of oxide materials for novel electronics by acquiring several new projects dedicated to the applied and fundamental research of gallium oxide as well as other oxide compounds intended to broaden the material basis. To support these studies, IKZ received in 2017 substantial EFRE funds to establish the “Applikationslabor für Materialien der Oxidelektronik” (Application Laboratory for Oxide Electronic Materials).

With the expertise on the growth of high purity, isotope - pure bulk germanium crystals, IKZ in 2017 joined the international LEGEND project, which provides an international frame for collaborative studies on the neutrinoless double beta decay of ^{76}Ge – a process, which may resolve some mysteries of particle physics.

Aside from the scientific advances, many achievements on the personnel level took place in 2017: the spin-off company from the IKZ GOLARES GmbH received the Founder Award of Leibniz Association, Dr. Kaspars Dadzis obtained the Young Scientist Award from the LIMTECH Alliance, and three of our doctoral students successfully defended their PhD works.

All of these would not happen without the dedication and expertise of our employees. Therefore, I would like to thank all colleagues at the IKZ who made these success stories in 2017 possible! I would also like to acknowledge the State of Berlin and the Federal Government for the financial support that makes the institute’s research programs possible.

Being supported by the research and development advances of the last seven years, the IKZ now prepares for the Leibniz Evaluation procedure taking place in December 2018. In the next year, we will also make the first steps to implement the institute´s new strategy. I am looking forward to cooperate with our internal and external colleagues and partners towards these exciting future challenges!

I wish you an enjoyable reading

Yours Sincerely,

Thomas Schröder

Content

Highlight

Classical Semiconductors

Dielectric & Wide Bandgap Materials

Layers & Nanostructures

Simulation & Characterization

Center for Laser Materials

The Institute

Photo: Lothar M. Peter © IKZ

Leibniz-Institut für Kristallzüchtung im Forschungsverbund Berlin e.V.

Founded 1992
Part of Forschungsverbund Berlin e.V.
Member of the Leibniz Association

The Institute

Das Leibniz-Institut für Kristallzüchtung (IKZ)

ist eine in Europa einzigartige Forschungs- und Service-Einrichtung, die sich experimentell und theoretisch mit den wissenschaftlich-technischen Grundlagen des Wachstums, der Züchtung, der Bearbeitung und der physikalisch-chemischen Charakterisierung von kristallinen Festkörpern beschäftigt. Dies reicht von der Grundlagenforschung bis hin zum Vorfeld industrieller Entwicklung. Die zurzeit entwickelten Materialien finden vorwiegend Verwendung in der Mikro-, Opto- und Leistungselektronik, der Photovoltaik, in Optik und Lasertechnik, in der Sensorik und Akustoelektronik.

Das Forschungsgebiet des IKZ umfasst Volumenkristalle, kristalline Schichten und Nanostrukturen sowie die Entwicklung von materialübergreifenden Kristallzüchtungstechnologien.

Arbeitsschwerpunkte des Institutes sind

- *Entwicklung von Züchtungs-, Bearbeitungs- und Charakterisierungsverfahren für Massivkristalle sowie kristalline Gebilde mit Abmessungen im Mikro- und Nanometerbereich sowie von materialübergreifenden Kristallzüchtungstechnologien*
- *Bereitstellung von Kristallen mit besonderen Spezifikationen für Forschungs- und Entwicklungszwecke*
- *Modellierung und Erforschung der Kristallwachstums- und Kristallzüchtungsprozesse*
- *Experimentelle und theoretische Untersuchungen zum Einfluss von Prozessparametern auf Kristallzüchtungsvorgänge und Kristallqualität*
- *Erforschung von Verfahren zur Kristallbearbeitung und der dabei ablaufenden Vorgänge*
- *Physikalisch-chemische Charakterisierung kristalliner Festkörper und Entwicklung geeigneter Methoden bis hin zur atomaren Ebene;*
  Aufklärung des Zusammenhangs zwischen Struktur und Eigenschaften kristalliner Materialien
- *Entwicklung und Bau von Anlagenkomponenten für die Züchtung, Bearbeitung und Charakterisierung von Kristallen*

Als Züchtungsverfahren werden Methoden der Züchtung aus der Schmelze, aus der Lösung, aus der Gasphase und davon abgeleitete Verfahren zur Herstellung kristalliner Schichten verwendet.

Durch die mögliche Synergie zwischen Volumenkristallzüchtung und der Abscheidung von Schichten verfügt das Institut über ideale Voraussetzung zur Herstellung von Substrat/Schicht-Kombinationen mit maßgeschneiderten Eigenschaften.

The Leibniz Institute for Crystal Growth

is a unique research and service institute in Europe, which is theoretically and experimentally investigating the scientific-technical fundamentals of crystal growth, processing and physico-chemical characterisation of crystalline solids. This ranges from explorative fundamental research to pre-industrial development. The materials presently in development are of fundamental importance in micro-, opto- and power electronics, in photovoltaics, in opto- and laser technology, in acousto-electronics and sensor technology as well as for fundamental research.

The research activities of the institute include bulk single crystals as well as crystalline layers and nanostructures, but also the development of comprehensive crystal growth technologies.

The research and service tasks of the institute include

- Development of technologies for growth, processing and characterization of bulk crystals and of crystalline structures with dimensions in the micro- and nanometer range and of comprehensive growth technologies
- Supply of crystals with non-standard specifications for research and development purposes
- Modelling and investigation of crystal growth processes
- Experimental and theoretical investigations of the influence of process parameters on crystal growth processes and crystal quality
- Development of technologies for the chemo-mechanical processing of crystalline samples and scientific investigation of related processes
- Physico-chemical characterisation of crystalline solids and development of suitable methods;
  investigation of the correlation between crystalline structures and properties
- Development and construction of components for growth, processing and characterization of crystals

Crystals are grown from the melt, from solutions and from the vapour phase and new techniques are developed and improved for the preparation of crystalline layers.

With the combination of bulk crystal growth and layer deposition, the institute possesses ideal conditions to produce customized substrate/layer-combinations.

The Institute

Materialien

- *Halbleiter mit großem Bandabstand (Oxide, Aluminiumnitrid) für Hochtemperatur-, Leistungs- und Optoelektronik*
- *Oxidische und fluoridische Kristalle für Lasertechnik, Optik, Sensorik und Akustoelektronik*
- *Silizium-Kristalle für Mikro- und Leistungselektronik und Photovoltaik*
- *Galliumarsenid für drahtlose Kommunikation und Hochfrequenztechnik*
- *Silizium/Germanium Kristalle für Strahlungsdetektoren und Beugungsgitter, kristalline Si/Ge-Schichten für thermoelektrische Anwendungen*
- *Silizium Schichten auf amorphen Unterlagen für die Photovoltaik*
- *Ferroelektrische und halbleitende Oxidschichten für die Mikro- und Leistungselektronik, Sensoren und Datenspeicher*

Materials presently in development

- Wide band gap semiconductors (aluminium nitride, oxides) for high temperature, power- and optoelectronics
- Oxide and fluoride crystals for acousto-electronics, laser-, opto- and sensor technology
- Silicon for power electronics and photovoltaics
- Gallium arsenide for wireless communication and high-frequency technology
- Silicon/germanium-crystals for radiation detectors and diffraction gratings, crystalline Si/Ge layers for thermoelectric devices
- Silicon layers an amorphous substrates for photovoltaics
- Ferroelectric and semiconducting oxide layers for micro- and power electronics, sensor applications or data storage

Zertifikat seit 2015
audit berufundfamilie

Das IKZ als familienfreundlicher Arbeitgeber

Das IKZ möchte seinen Beschäftigten ein offenes, kooperatives und familienfreundliches Arbeitsumfeld bieten. Das Institut unterstützt daher seine Mitarbeiterinnen und Mitarbeiter bei der Vereinbarkeit von Arbeit und Familie, z.B. durch flexible Regelungen zur täglichen Arbeitszeit oder durch variable Regelungen zu Teil- und Vollzeitbeschäftigung.

Seit 2015 ist das Institut zertifiziert durch das audit berufundfamilie. Damit verbunden hat es Ziele einer familienbewussten Personalpolitik definiert und sich verpflichtet. In den folgenden drei Jahren haben wir die in diesem Prozess definierten Maßnahmen umgesetzt. Das Audit wird 2018 wiederholt.

Das audit steht unter der Schirmherrschaft der Bundesfamilienministerin und des Bundeswirtschaftsministers, nähere Informationen finden sich unter www.beruf-und-familie.de

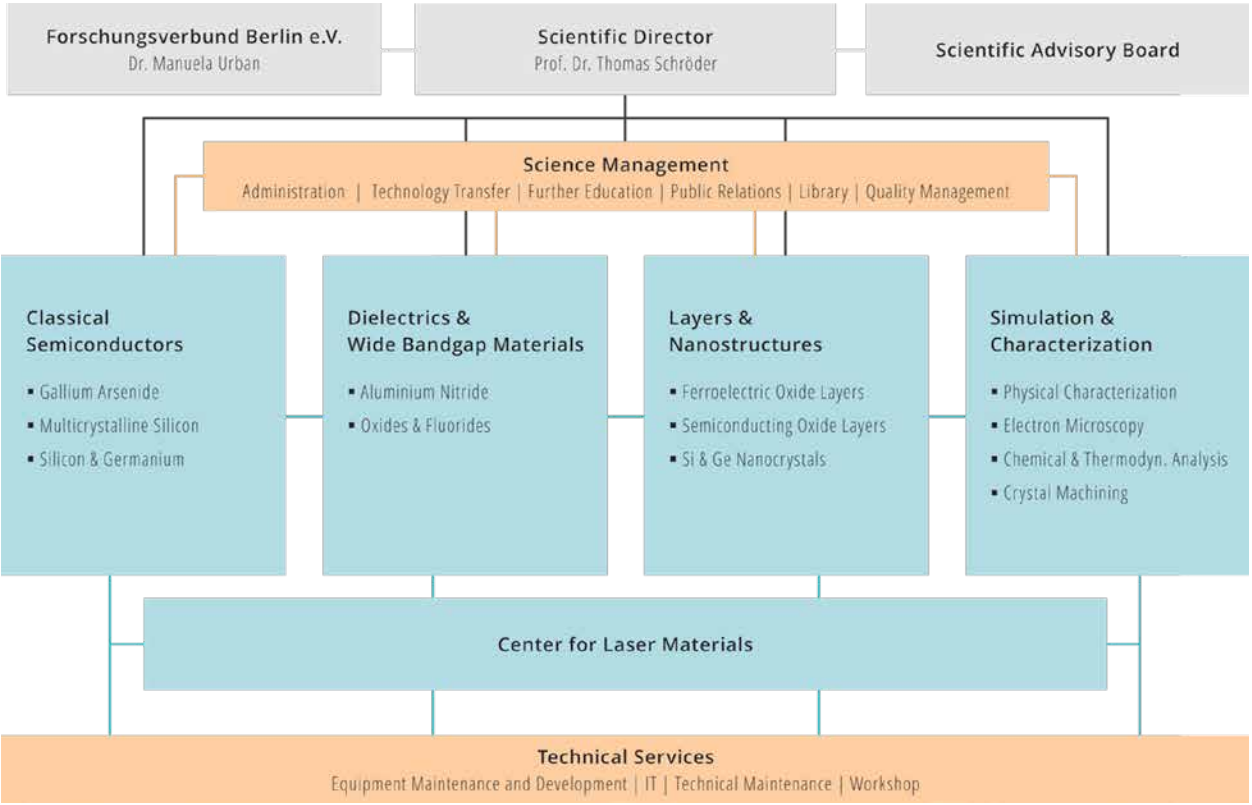
IKZ as family-friendly employer

The institute intends to create a co-operative and open working environment for all employees. It places special emphasis on the reconcilability of job and family, offering flexible working time models as well as full or part-time employments.

In 2015, the institute has been awarded the *audit berufundfamilie* certificate for its family-friendly human resources policy. During the following three years, we have been implementing the objectives defined in this process. The audit will be repeated in 2018.

The certificate is issued under the auspices of the German Federal Minister for Families and the German Federal Economics Minister. More information is available under www.beruf-und-familie.de

Organisation Chart / Organigramm



Scientific Advisory Board 2017
Wissenschaftlicher Beirat 2017

apl. Prof. Dr. -Ing. Michael Heuken (chair)
Faculty of Electrical Engineering and Information Technology, RWTH Aachen University & Vice President of Research and Development AIXTRON SE, Aachen

Dr. Lothar Ackermann
Forschungsinstitut für mineralische und metallische Werkstoffe, Edelsteine/Edelmetalle – FEE GmbH, Idar-Oberstein

Dr. Hubert Aulich
SC Sustainable Concepts GmbH, Erfurt

Prof. Dr. Saskia Fischer (vice chair)
Department of Physics, Humboldt-Universität zu Berlin

Prof. Dr. Michael Kneissl
Institute of Solid State Physics, Technische Universität Berlin

Prof. Dr. Götz Seibold
Brandenburgische Technische Universität Cottbus-Senftenberg

Dr. Martin Strassburg
Osram Opto Semiconductors GmbH, Regensburg

Representative of the State of Berlin

Dr. Björn Maul
The Governing Mayor of Berlin
Senate Chancellery – Higher Education and Research

Representative of the Federal Republic

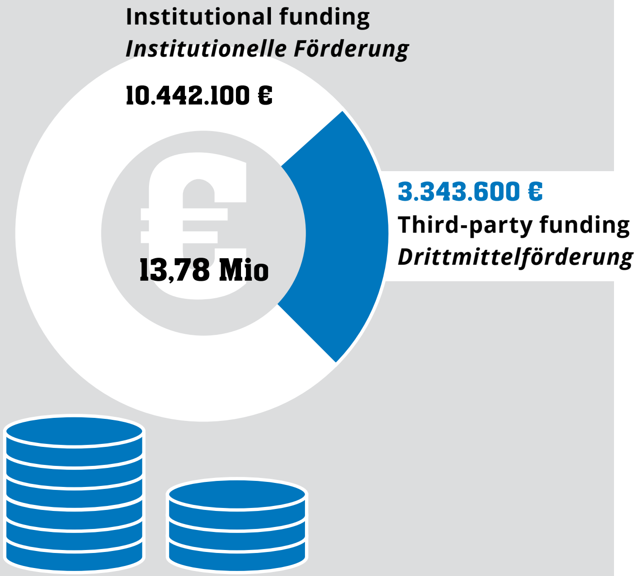
Dr. Anne Parge
Bundesministerium für Bildung und Forschung, BMBF Bonn / Berlin

The Institute

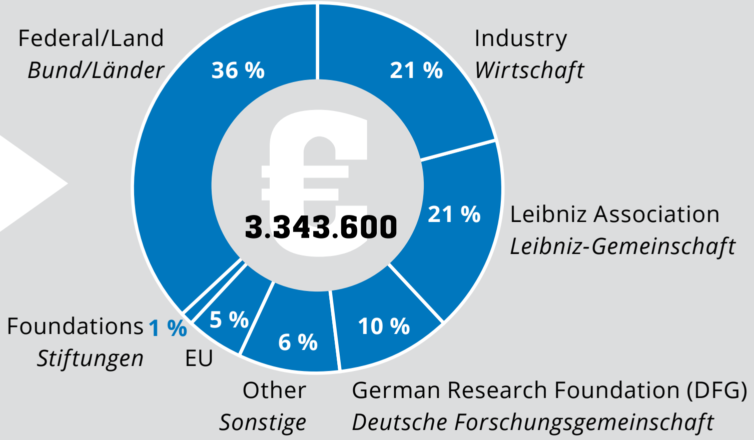
2017 in figures 2017 in Zahlen

Budget

Total Gesamt



Third-party funding Drittmittelförderung



Education Lehre

5

Master and Bachelor projects completed.
Abgeschlossene Master- und Bachelorarbeiten.

18

Running PhD projects.
3 PhD theses were defended.
Laufende Promotionsprojekte.
3 Dissertationen wurden verteidigt.

Publications Publikationen

79

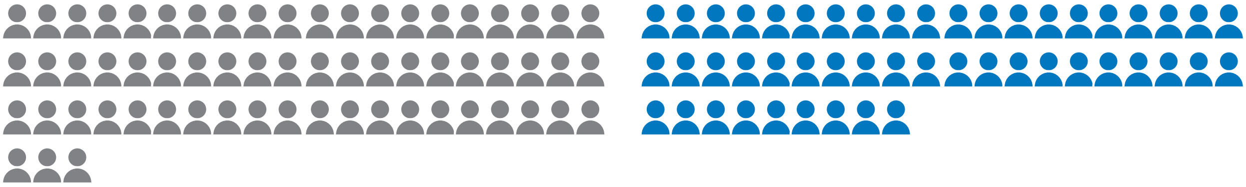
Published peer-review scientific articles.

Artikel in referierten (peer-review) Journalen.

The Institute

Staff total Personal gesamt*

112

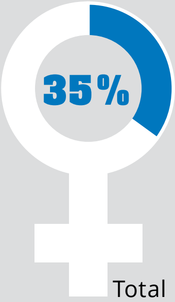


56 % Scientific employees
Wissenschaftliche Mitarbeiter/innen

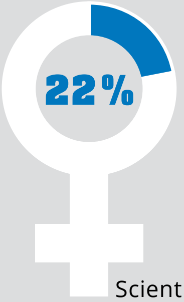
44 % Infrastructure personnel
Infrastrukturpersonal

* not including Bachelor-/Master students and student assistants.
ohne Bachelor-/Masterstudenten und studentische Hilfskräfte.

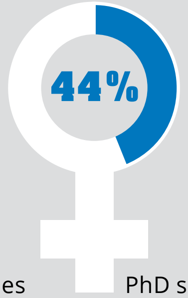
Female proportion Frauenanteil



Total
Gesamt

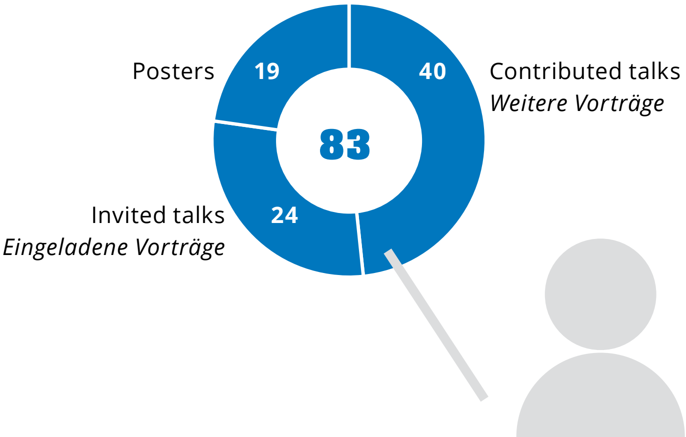


Scientific employees
Wissenschaftliche Mitarbeiterinnen



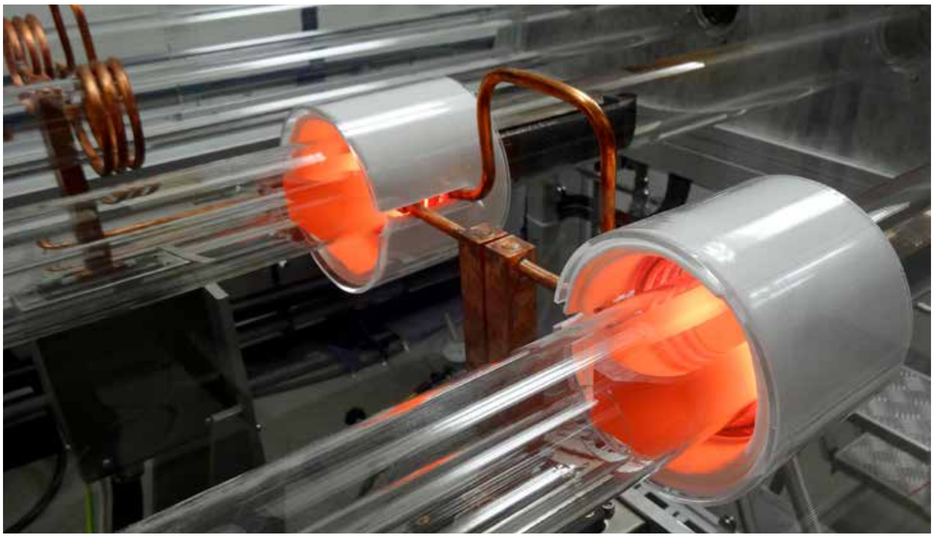
PhD students
Doktorandinnen

Contributions in international conferences
Beiträge auf internationalen Konferenzen



Timeline: Overview of 2017 in IKZ
Chronik: Überblick 2017 im IKZ

Photo: ©IKZ



Multizonenreinigung (MZR) für Germanium

Zone refining furnace for germanium purification

Januar

Hochreines Germanium für die Aufklärung des Rätsels um das Neutrino

Zum Beginn des Jahres ist unser Institut der internationalen Forschungskooperation LEGEND beigetreten. Ziel des gemeinsamen Vorhabens ist es, ein extrem seltenes Ereignis zu beobachten: den neutrinolosen Doppelbetazerfall von Germanium-76, der laut Theorie beweisen könnte, dass Neutrinos ihre eigenen Antiteilchen sind. Diese Eigenschaft könnte den bislang ungeklärten Überschuss von Materie (gegenüber der Antimaterie) im Universum erklären.

47 Forschungseinrichtungen weltweit forschen an den verschiedenen Aspekten des Projekts, wie z.B. der Verbesserung der Abschirmung des Experiments, der Erforschung strahlungsarmer Materialien oder der Verbesserung der Empfindlichkeit der Signalerkennung. Die Aufgabe des IKZ ist es, eine Technologie für die Züchtung von Einkristallen mit niedriger Versetzungsdichte aus mit dem Isotop ^{76}*Ge angereicherten Germanium. Diese dienen im Experiment sowohl als Strahlungsquelle als auch als Detektormaterial.*

Theoretische Studien sagen weniger als einen möglichen Zerfall pro Jahr und Kilogramm Detektormaterial voraus. Aufgrund dieser geringen Wahrscheinlichkeit benötigt das Experiment insgesamt 500 Detektoren mit jeweils 2 kg dieser angereicherten Germaniumkristalle.

January

High-purity germanium to uncover the mystery of neutrino

At the beginning of the year, our institute joined the international collaboration LEGEND. The goal of the experiment is to register the extremely rare event – the neutrinoless double-β decay of germanium-76, which, according to the theory, will prove that neutrinos are their own antiparticles and thus explain the excess of matter in the universe.

47 international research centres are working on the various aspects of the project such as enhancing the experiment shielding, researching low-radiation materials or improving the sensitivity of signal detection. The mission of the IKZ is to develop the growth process to obtain bulk germanium ^{76}Ge-enriched crystals with low dislocation density, which will serve both as a radiation source and as a detector in the experiment.

Theoretical studies predict less than one decay per year per kilogram of detector material. Due to this rarity, the experiment will require in total 500 detectors of 2 kg enriched germanium crystals.

Overview

März

Ausgezeichnet beschichten – Gründerpreis für IKZ-Ausgründung GOLARES

Am 24. März erhielt die GOLARES GmbH – das Spin-off Unternehmen aus dem IKZ – den Gründerpreis der Leibniz-Gemeinschaft.

GOLARES bietet die Technologie, um Oberflächen von elektronischen und optoelektronischen Komponenten mittels Gasplasmaabscheidung zu beschichten. Das Angebot richtet sich vorwiegend an kleine und mittlere Unternehmen. Die Firmengründer Dr. Michael Arens und Dr. Sebastian Golka haben ein Verfahren entwickelt, das eine geringe Oberflächenschädigung erlaubt und es ermöglicht, Schichten mit höchster Qualität zu erzielen.

Mit dem Preisgeld von 50.000 € der Leibniz-Gemeinschaft erhalten die beiden Gründer die Möglichkeit, den Markteintritt erfolgreich zu gestalten und damit ihr Unternehmenskonzept weiter auszubauen.

March

Quality coatings for electronics – Founders Award for IKZ spin-off

On 24th of March, The GOLARES GmbH – the spin-off company from IKZ – received the Founder Award of Leibniz Association.

GOLARES provides the technology to coat surfaces of electronic and optoelectronic components with gas plasma for small and medium-sized companies. The founders of the company Dr. Michael Arens and Dr. Sebastian Golka have developed a method that has a low risk of creating surface damage and allows to deposit films with superior quality.

With the prize of € 50.000 from the Leibniz Association, the two founders have the opportunity to successfully enter the market and thereby further expand their company concept.

Photo: Tina Merkau © WISTA-Management GmbH

Die Preisträger: Dr. M. Arens (rechts) & Dr. S. Golka (links)

The award winners: Dr. M. Arens (right) & Dr. S. Golka (left)

April

Gründung des Zentrums für Lasermaterialien

Mit einem zweitägigen Workshop startete die neue Forschungseinheit offiziell ihre Arbeiten am IKZ. Renommierte Experten aus Wissenschaft und Industrie trafen sich zum wissenschaftlichen Austausch und zur Diskussion der zukünftigen Herausforderungen des Zentrums für Lasermaterialien, sowie der möglichen industriellen Anwendungsbereiche.

Mehr über das Zentrum finden sich als Highlight auf Seite 18.

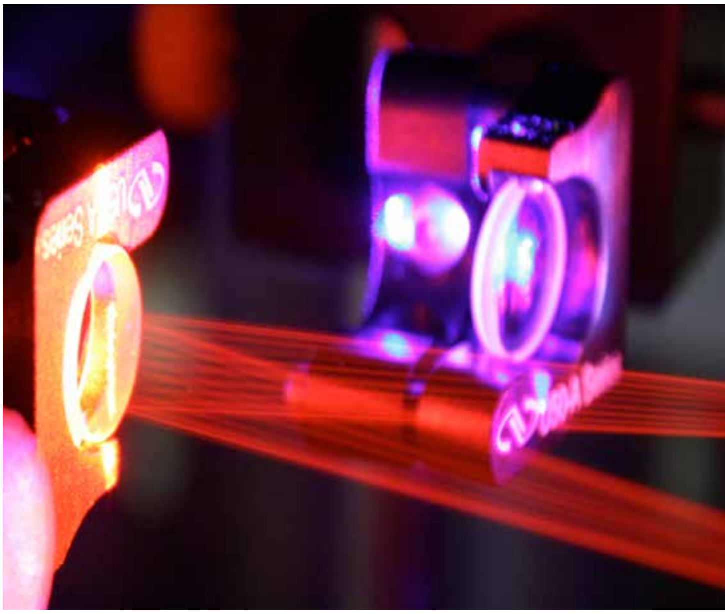


Photo: Christian Kränkel ©IKZ

April

Launching the Center for Laser Materials

A two-day workshop set the official start of the works in the new research unit at the IKZ. Renowned experts from the scientific and industrial community met to exchange current competencies, discuss prospective challenges of the novel Center for Laser Materials, and possible industrial applications.

Read more about the new Center in the Highlights section on the page 18.

Mai

25 Jahre Forschungsverbund: eine Feier der Verbundenheit

Auf Empfehlung des Wissenschaftsrats wurden vor 25 Jahren aus Teilen der Akademie der Wissenschaften neue Forschungseinrichtungen gegründet, darunter auch das IKZ. Die wissenschaftliche Expertise war vorhanden, nicht aber Erfahrungen mit der Verwaltung. Und schließlich mussten Arbeitsverträge geschlossen und Ausrüstungsgegenstände beschafft werden. Als Übergangslösung wurde damals der Forschungsverbund Berlin gegründet, der diese Kompetenzen für insgesamt acht Institute bereit stellen sollte. Ursprünglich nur als Übergangslösung für fünf Jahre gedacht, feierte der Forschungsverbund am 18. Mai 2017 nun sein 25-jähriges Bestehen.

Photo: Volkmar Otto © Forschungsverbund Berlin e.V.

G. Wagner während der FVB-Jubiläumsfeier in der Urania.
G. Wagner during FVB celebration event in the Urania.

In einer Festveranstaltung in der Urania hatte das interessierte Publikum die Gelegenheit, sich über die Arbeit der einzelnen Institute zu informieren - von der Zoo- und Wildtierforschung bis zur Kristallzüchtung. Neben Vorträgen wie dem von Günter Wagner über die Bedeutung der Kristallzüchtung in der modernen Gesellschaft kam auch der wissenschaftliche Nachwuchs zu Wort. Owen Ernst erklärte in dem Science Slam Wettbewerb der Institute was passiert, wenn die Sonne auf Steine scheint.

May

25 years Forschungsverbund Berlin: a celebration of unity.

25 years ago, on the recommendation of the Council of Science and Humanities, new research institutions – including the IKZ – were founded from the parts of the Academy of Sciences. The scientific expertise was at hand, but the administration experience was missing. After all, employment contracts had to be signed and equipment had to be procured. As an interim solution, the Forschungsverbund Berlin was founded at that time to provide support with recruitment procedures, equipment procurement and other administrative tasks for a total of eight institutes. Originally intended only as a temporary solution for five years, the Forschungsverbund celebrated its 25th anniversary on 18 May 2017.

In a festive event that took a place in the Urania, the interested public had the opportunity to inform themselves about the work of the individual institutes - from Zoo and Wildlife Research to Crystal Growth. In addition to lectures, such as Günter Wagner's on the importance of crystal growth for modern society, there was also a chance for young scientists to contribute. In the Science Slam competition, the doctoral student of the IKZ Owen Ernst explained what happens when the sun shines on stones.

Photo: ©IKZ

Juli

Austausch und Zusammenarbeit: Fünftägiger Workshop zur Kristallzüchtungstechnologie in der grünen Umgebung von Potsdam

Im Juli fand der internationale Workshop zur Kristallzüchtungstechnologie (International Workshop on Crystal Growth and Technology, IWCGT-7) im idyllischen Umfeld von Potsdam statt.

Der Workshop wurde vom Leiter der Abteilung Dielektrika & Wide Bandgap Prof. Matthias Bickermann mit Unterstützung von weiteren Mitarbeiter/innen des IKZ organisiert und war der siebte in der 1998 von Hans Scheel, einem Pionier der Kristallzüchtung, organisierten Reihe.

Ziel des Treffens ist es, die internationalen Experten der Kristallzüchtung zusammenzubringen und eine Plattform zwischen der akademischen Forschung und der industriellen Entwicklung auf diesem Gebiet zu schaffen. Diese Gelegenheit wurde von 99 Teilnehmern aus insgesamt 19 Ländern auch in diesem Jahr wieder gerne genutzt. Der nächste Workshop wird im Jahr 2020 im Berliner Umland stattfinden.

July

Exchange and collaboration: five-day workshop on crystal growth technology in the green outskirts of Potsdam

In July, the International Workshop on Crystal Growth and Technology (IWCGT-7) took place at an idyllic venue near the lake in Potsdam.

The workshop was organized by the head of Dielectrics & Wide Bandgap Materials department Prof. Matthias Bickermann with the support of other members of the IKZ and became the 7th in the series initiated in 1998 by one of the pioneers of crystal growth Hans Scheel.

The goal of the meeting is to bring together the international experts in crystal growth technology and build a bridge between the academic research and the industrial development in this field. In 2017, 99 participants from 19 countries gladly seized this opportunity. The next workshop will be held in 2020 in the surroundings of Berlin.

September

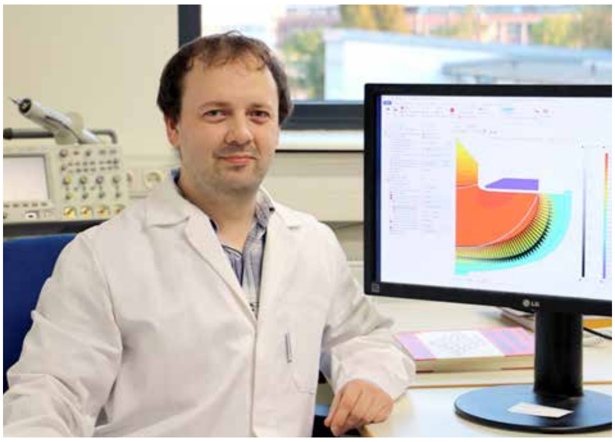


Photo: ©IKZ

Dr. Kaspars Dadzis

Die nächst Generation von Kristallzüchtungs-Experten

2017 erhielt Dr. Kaspars Dadzis den Young Scientist Award der LIMTECH Alliance – der Initiative zur Unterstützung der Forschung auf dem Gebiet der Flüssigmetalltechnologien.

Dr. Dadzis ist spezialisiert auf die Modellierung und Simulation im Bereich der Kristallzüchtung und verfügt über langjährige Berufserfahrung in der Wissenschaft sowie in der Kristallzüchtungsindustrie. Im Jahr 2016 trat er in die Arbeitsgruppe „Silizium & Germanium" am IKZ ein und beschäftigt sich seitdem mit der Entwicklung neuer Methoden zur Züchtung kristalliner Materialien.

September

Next generation of crystal growth professionals

Dr. Kaspars Dadzis has received the Young Scientist Award from the LIMTECH Alliance – the initiative that supports the research in the field of liquid metal technologies.

Dr. Dadzis specializes on the model experiments and simulations in crystal growth and has long working experience in academia as well as in crystal growth industry. In 2016, he joined the "Silicon & Germanium" working group at the IKZ and since then has been working on the development of new methods for the growth of crystalline materials.

November

Galliumoxid zeigt seine Stärke für die Leistungselektronik

In den letzten Jahren ist das Interesse an dem Halbleitermaterial Galliumoxid (β-Ga_2O_3) aufgrund seines hohen Potenzials für elektronische Hochleistungsanwendungen gestiegen. In 2017 gelang es sowohl dem Air Force Research Laboratory (AFRL) in Ohio, USA, als auch dem Ferdinand-Braun-Institut, Leibniz-Institut für Höchstfrequenztechnik (FBH) in Berlin, die ersten Prototypen eines Metall-Isolator-Halbleiter-Feldeffekttransistors (MISFETs) auf der Basis von am IKZ gewachsenen Galliumoxid-Epitaxieschichten herzustellen. Die Transistoren wiesen gute Bauelementeigenschaften auf und zeigten damit das große Potenzial von β-Ga_2O_3 für die Leistungselektronik.

Aufgrund dieser vielversprechenden Ergebnisse wurden die Arbeiten zu diesem Thema im Rahmen von mehreren neuen Forschungsprojekten fortgeführt. Dazu wurde am 1. November unter Leitung von Dr. Günter Wagner das auf drei Jahre angelegte Projekt „Oxikon" in Kooperation mit dem FBH und der Technischen Universität Berlin gestartet, das vom BMBF im Rahmen des VIP+-Programms gefördert wird.

Mehr Informationen zu den laufenden Forschungsprojekten erfahren Sie in den Berichten der Gruppen „Oxide & Fluoride" und „Halbleitende Oxidschichten".

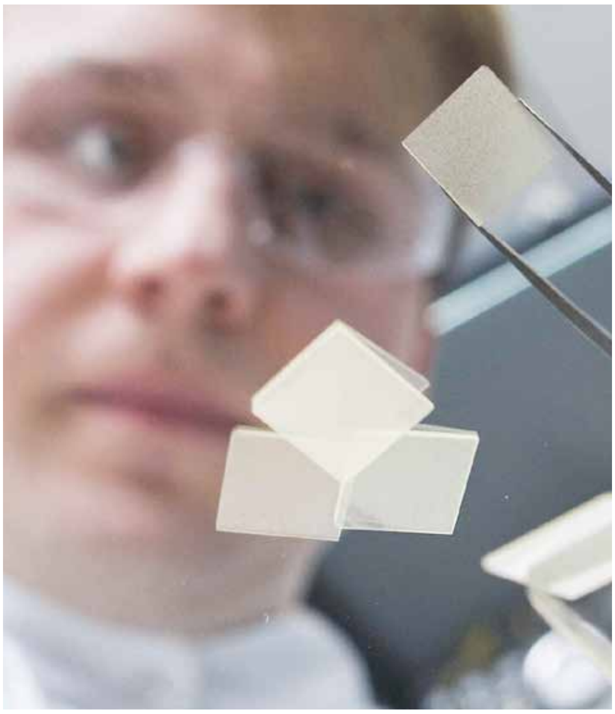


Photo: Volkmar Otto © Forschungsverbund Berlin e.V.

November

Gallium oxide for power electronics strengthens its positions

In the last years, the interest in β-Ga_2O_3 semiconductors has boosted due to its high potential for high-power electronic applications. In 2017, the Air Force Research Laboratory (AFRL) in Ohio, USA and Ferdinand-Braun-Institute (FBH) in Berlin, Germany have both successfully presented prototypes of the metal-insulator-semiconductor field effect transistors (MISFETs) based on β-Ga_2O_3 substrates and homoepitaxial layers grown at the IKZ. The transistors have competitive component properties and showed the great potential of β-Ga_2O_3 for power electronics applications.

These results laid the basis for the further investiations on β-Ga_2O_3-based devices for power electronics, and IKZ has launched several new collaboration projects. In particular, on the 1st of November started a joint three-year long project "Oxikon" between IKZ, Ferdinand-Braun-Institute and Technische Universität Berlin funded in the frame of the BMBF VIP+ program. Günter Wagner, the head of the group "Semiconducting Oxide Layers", is in charge of the project management.

To learn more about the on-going research projects on oxide semiconductors for power applications, see in the reports from the groups "Oxides & Fluorides" and "Semiconducting Oxide Layers".

Dezember

Ein neuer Direktor für das IKZ

Anfang Dezember nahm Prof. Dr. Thomas Schröder den Ruf auf die Professur „Kristallwachstum" an der Humboldt-Universität zu Berlin an, die mit der Position des Direktors des IKZ verbunden ist.

Damit löst er Prof. Dr. Günther Tränkle, Direktor des Ferdinand-Braun-Instituts für Höchstfrequenztechnik, ab, der seit 2013 das Institut als Interimsdirektor geleitet hatte. Prof. Dr. Schröder wird seine Funktion am IKZ Anfang 2018 offiziell übernehmen.

December

Photo: © WISTA-Management GmbH

Appointment of the new head of the IKZ

In the beginning of December Prof. Dr. Thomas Schröder accepted the call for professorship "Crystal Growth" at Humboldt-Universität zu Berlin which is associated to the position of the director of the IKZ.

With this appointment, he takes over from Prof. Dr. Günther Tränkle, Director of the Ferdinand-Braun-Institut für Höchstfrequenztechnik, who had headed the institute as an interim director since 2013. Prof. Dr. Schröder will officially assume his position at the IKZ in the beginning of 2018.

Aufbau des Zentrums für Lasermaterialien am IKZ:

Interview mit Christian Kränkel und Elena Castellano-Hernández

Im August 2016 bewilligte das BMBF das Projekt EQuiLa – Forschung und Qualifizierung innovativer Lasermaterialien und Kristalle. Im Rahmen dieses Projektes erhielt das Leibniz-Institut für Kristallzüchtung die Möglichkeit, das neue Zentrum für Lasermaterialien (ZLM) zu etablieren. Anfang 2017 übernahm Dr. habil. Christian Kränkel die Leitung des neuen Forschungsbereichs im IKZ. In diesem Interview diskutieren wir mit Dr. Kränkel und der ersten Doktorandin des ZLM, Elena Castellano-Hernández, über die neue Abteilung und ihre Ziele, was ihr Interesse an der Laserphysik weckte und warum sie sich für das IKZ entschieden haben.

Warum habt ihr euch für Laser als Forschungsgebiet entschieden? Was interessiert euch persönlich daran?

Christian: *Eigentlich war es eher ein Zufall, dass ich bei den Lasern landete. Zu Beginn meines Studiums habe ich mich für Mineralogie und Physik eingeschrieben und dann aber nur Physik studiert. Während meines Studiums in Hamburg habe ich eine ausgezeichnete Vorlesung in Laserphysik bei Günter Huber gehört, die mich motivierte, mich auf diesem Gebiet zu spezialisieren. Ich fand, dass die Laserphysik eine perfekte Mischung aus angewandter Physik - in Bezug auf das Arbeiten mit den Händen – und theoretischer Physik – in Bezug auf die Arbeit mit dem Kopf - ist.*

Später habe ich in der Gruppe von Günter Huber über Ytterbium-dotierte Materialien für Ultrakurzpuls-Laser promoviert und anschließend zwei Jahre lang an der ETH Zürich als Postdoc in der Forschungsgruppe von Ursula Keller gearbeitet. Danach hatte ich die Chance, im Rahmen der Exzellenzinitiative als Nachwuchs-Gruppenleiter nach Hamburg zurückzukehren. Dort habe ich mich im Februar 2017 habilitiert.

Elena: *Ich habe mich im dritten Jahr meines Bachelorstudiums der Physik an der Universidad Autónoma de Madrid für Optik und Photonik interessiert. Damals begann ich, Vorträge zu diesem Thema zu hören – ich war fasziniert vom Verhalten des Lichts und wollte mehr wissen. Als ich meinen Master-Abschluss in Physik und Materialtechnik an der Universidad del País Vasco machte, suchte ich aktiv nach Positionen in der Laserphysik. Nach meiner Masterarbeit arbeitete ich 3,5 Jahre am Instituto de Ciencia de Materiales de Madrid in einer Laserforschungsgruppe.*

Wie seid ihr auf das IKZ aufmerksam geworden? Warum habt ihr euch entschieden, zum IKZ zu kommen, um diese Forschung zu betreiben?

Christian: *Ich habe das erste Mal vom IKZ gehört, als ich an der Universität Hamburg meine Masterarbeit über Ytterbium-dotierte Yttriumvanadat-Kristalle ($Yb:YVO_4$) schrieb. Die Kristalle wurden am IKZ von Margitta Bernhagen gezüchtet. Seitdem stand ich in einem regen Austausch mit Leuten aus dem Institut, insbesondere mit Reinhardt Uecker, dem ehemaligen Leiter der Gruppe „Oxide & Fluoride“. Ich profitierte auch von verschiedenen Charakterisierungsmethoden am IKZ, die wir in Hamburg nicht hatten, und von Berechnungen für das Kristallwachstum von Detlef Klimm.*


Photo: Tina Merkau ©IKZ

Dr. Christian Kränkel – head of the Center for Laser Materials.
Dr. Christian Kränkel - Leiter des Zentrums für Lasermaterialien.

Eigentlich war das für mich schon immer ein sehr interessantes Forschungsgebiet. Und dann hatte Günter Tränkle (Red: kommissarischer Direktor des IKZ zu dieser Zeit) eine sehr gute Gelegenheit, Mittel für das Zentrum für Lasermaterialien einzuwerben, und zu diesem Zeitpunkt hatte ich gerade meine Habilitation in Hamburg abgeschlossen. Es war der perfekte Zeitpunkt für mich, meine Position noch einmal zu wechseln und meine Forschung am IKZ in Berlin fortzusetzen.

Obwohl ich hier stark von der bestehenden Infrastruktur profitiere, bin ich natürlich nicht allein im Zentrum, sondern brauche Leute, die mit mir arbeiten – und so wurde Elena die erste Doktorandin am ZLM.

Elena: *Im Jahr 2015 erhielt ich ein Stipendium der spanischen Regierung für einen kurzen Forschungsaufenthalt. Ich wählte Christians Gruppe in Hamburg und verbrachte dort 4 Monate. Das war eine sehr gute Erfahrung, so dass ich 2016, als ich den gleichen Zuschuss erhielt, wieder dorthin ging. Damals fragte ich Christian nach der Möglichkeit, mich bei seiner Gruppe zu bewerben. Als er mir im Januar 2017 eine Doktorandenstelle am IKZ anbot, zögerte ich nicht.*

Establishing the Center for Laser Materials at the IKZ:

Interview with Christian Kränkel and Elena Castellano-Hernández

In August 2016, the BMBF approved the EQuiLa project – research and qualification of innovative laser materials and crystals. Within the scope of this project, the Leibniz-Institut für Kristallzüchtung got the possibility to expand and establish the new Zentrum für Lasermaterialien (Center for Laser Materials, ZLM). At the beginning of 2017, Dr. habil. Christian Kränkel was hired to take the lead in the new research area in the IKZ. In this interview, we discuss with Dr. Kränkel and the first PhD student of the ZLM, Elena Castellano, the new department and its goals, what sparked their interest in laser physics, and why they chose IKZ for their research.

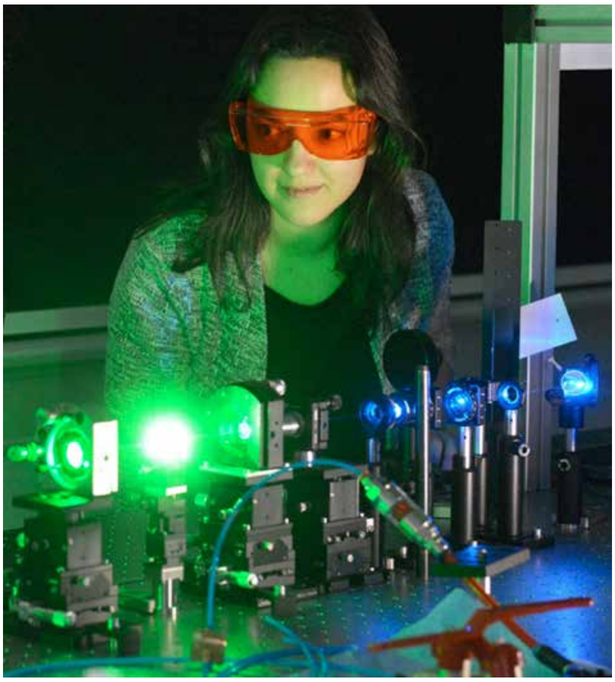


Photo: Tina Merkau © IKZ

Elena Castellano-Hernández investigates green emitting terbium-doped laser.
Elena Castellano-Hernández untersucht grün emittierende Terbium-dotierte Laser.

Why did you choose lasers as the area of your research? What interests you personally in it?

Christian: Actually, it was more of a coincidence that I ended up in lasers. When I started to study, I signed up for mineralogy and physics. As I got a place to study in physics, I resigned from mineralogy. When I studied physics in Hamburg, I heard an excellent lecture in laser physics held by Günter Huber which motivated me to specialize in this field. I found laser physics to be a perfect mix between applied physics – in terms of using your hands, and theoretical physics – in terms of using your brain.

Later, I did my PhD thesis in the group of Günter Huber on ytterbium-doped materials for ultrashort pulsed lasers, and then I worked for two years at the ETH Zürich as a postdoc in the research group of Ursula Keller. After that, I had a good opportunity to come back to Hamburg in the framework of the Excellence Initiative and to be a Junior Group Leader. There I received my habilitation degree in February 2017.

Elena: I became interested in optics and photonics in the third year of my bachelor in physics at the Universidad Autónoma de Madrid. It was then when I started to have lectures on this topic – I was fascinated with the behaviour of light and I wanted to know more. When I finished my Master degree in Physics and Material Technology at the University of the Basque Country, I actively looked for positions in laser physics. After my master thesis, I worked for 3.5 years at the Instituto de Ciencia de Materiales de Madrid, in a laser research group.

How did you learn about the IKZ? Why did you decide to come to IKZ to do this research?

Christian: I first learned about IKZ when I did my master thesis on ytterbium-doped yttrium vanadate (Yb:YVO$_4$) crystals at the Universität Hamburg. The crystals were grown at the IKZ, by Margitta Berghagen, and ever since then, I was in a vivid exchange with people from the institute, particularly with Reinhardt Uecker, the former leader of the “Oxides & Fluorides” group. I also benefited from various characterization methods at the IKZ that we did not have in Hamburg and calculations for crystal growth done by Detlef Klimm.

Actually, that was always a very interesting research area for me. Later it turned out that Günter Tränkle (Ed: acting director of the IKZ at the time) had a very good opportunity to obtain funding for the Center for Laser Materials, and at this time I had just received my habilitation degree in Hamburg. It was perfect timing for me to change my affiliation again and come to Berlin to continue my research at the IKZ.

Although I strongly benefit from the existing infrastructure here, of course, the Center is not just me, I need people working with me – and so Elena became the first PhD student at the ZLM.

Elena: In 2015 I received a grant from the Spanish government to make a short research stay. I chose Christian’s group in Hamburg and spent 4 months there. The experience was very good, so when in 2016 I got the same grant I went there again. At that time I asked Christian about the possibility to enrol in his group. When in January of 2017 he offered me a PhD position at IKZ, I did not hesitate.

Was ist der Vorteil des Zentrums für Lasermaterialien im IKZ?

Christian: *Das Zentrum für Lasermaterialien ist eine logische Fortsetzung der Wertschöpfungskette der Forschung am IKZ. Das IKZ verfügt über ein enormes Know-how im Bereich der Kristallzüchtung, und Laser sind eine der möglichen Anwendungen von Kristallen. Es gibt eine Vielzahl von Kristallen, die für Laser verwendet werden könnten, aber man wird kaum ein Unternehmen finden, das viele verschiedene Kristalle züchten kann, um zu testen, ob diese für ihre spezielle Laseranwendung geeignet sind. Das ist die Forschung, die in einem Institut für Kristallzüchtung durchgeführt werden muss. Es ist nur logisch, meine Forschungen zu Lasern und zur spektroskopischen Charakterisierung dieser Materialien hier am IKZ zu platzieren.*

Elena: *Die Zusammenarbeit innerhalb des Instituts ist sehr wichtig. Wir konzipieren die Materialien, die für die Entwicklung unserer Laser geeignet sind, dann züchten unsere Kollegen sie, und danach charakterisieren wir die Kristalle und konstruieren die Laser. Es ist also ein sehr interaktiver Prozess.*

Christian: *Und es ist schnell! Das Feedback an die Züchter ist viel schneller, als wenn die Zusammenarbeit zwischen zwei verschiedenen Instituten ablaufen würde.*

Gibt es noch andere ähnliche Einrichtungen wie das Zentrum für Lasermaterialien auf der Welt?

Christian: *Tatsächlich ist es sogar schwierig, ein Forschungszentrum ähnlich dem IKZ irgendwo auf der Welt zu finden. Die Erweiterung der Kompetenzen im Bereich der Laser und Kristalle für optische Anwendungen macht es nun noch einzigartiger. Ich glaube nicht, dass es auf meinem Forschungsgebiet ein anderes Institut gibt, das über vergleichbare Kenntnisse und eine vergleichbare Forschungsinfrastruktur verfügt.*

Elena: *In vielen Lasergruppen züchten die Doktoranden auch die Kristalle, aber sie sind keine Experten. Dies erfordert viel Zeit während der Promotion. Die Situation hier ist viel besser. Wir haben erfahrene Kristallzüchter, die für diesen Teil der Arbeit verantwortlich sind, so dass man sich auf seine eigene Forschung konzentrieren kann.*

Habt ihr zum ersten Mal ein Labor von Grund auf eingerichtet?
Welchen Herausforderungen seid ihr dabei begegnet?

Christian: *Ich habe bereits einmal Labore eingerichtet, als ich meine Gruppe in Hamburg gegründet habe, aber damals hatte ich zumindest schon einen optischen Tisch, also habe ich nicht von Null angefangen. Hier fing es mit einem Labor an, das für einen anderen Zweck ausgestattet war und geleert und renoviert werden musste, auch alle Wasserleitungen mussten neu verlegt werden. Das war eine neue Erfahrung. Außerdem habe ich einen optischen Tisch gekauft, der etwa 3x4 Meter groß und rund zwei Tonnen schwer ist. Der musste mit einem Kran in den zweiten Stock gebracht werden. Das war eine neue Erfahrung, selbst für die Menschen, die die Tische geliefert haben.*

Außerdem war es neu für mich, der aus dem universitären Umfeld kommt, zu sehen, wie gut der technische Service hier am IKZ auf diese Art von Arbeit vorbereitet war. Vor allem Jens Klose und seine Leute unterstützten uns sehr. Das hat gut funktioniert.

Jetzt kämpfen wir natürlich immer noch mit den Ausschreibungsregeln der Stadt Berlin für den Kauf von teuren Geräten, aber trotzdem, denke ich, sind wir auf einem guten Weg - wir haben ein funktionierendes Labor mit vielen modernen Geräten. Das ist der angenehme Teil der Forschung.

Elena: *Für mich war es eine neue Erfahrung, die ganze Ausrüstung zu kaufen. Das ist sehr interessant, weil man eine Vorstellung vom Preis der Dinge bekommt, mit denen man arbeitet. Man lernt viel über die Geräte, die man benutzt, weil man alle Eigenschaften überprüfen muss, bevor man sich für einen Kauf entscheidet. Außerdem ist es toll, wenn man die Möglichkeit hat, alles nach seinen Bedürfnissen und Vorlieben aufzubauen.*

Photo: E. Castellano © IKZ

Photo of the ZLM laser and spectroscopy laboratory in March (left) and December 2017 (right).

Foto des Laser- und Spektroskopielabors des ZLM im März (links) und Dezember 2017 (rechts).

What is the advantage of having the Center for Laser Materials in IKZ?

Christian: The Center for Laser Materials is a logical prolongation of the value chain of the research at the IKZ. The IKZ has enormous knowledge in the growth of crystals, and lasers are one of the possible applications of crystals. There is a variety of crystals that could be used for lasers, but you will hardly find a company that can grow many different crystals to test them whether they are suitable for their particular laser application. This is the research that has to be done in an institute devoted to crystal growth. It's only logical to place my research on the laser and spectroscopic characterization of these materials here at the IKZ.

Elena: The collaboration inside the institute is very important. We design the materials that are suitable for developing our lasers, then our colleagues grow them, and after that, we characterize the crystals and build the lasers. So, it's a very interactive process.

Christian: And it's fast! The feedback loops are much shorter than if that would be between two different institutes.

Is there other analogies of the Center for Laser Materials in the world?

Christian: Actually, it's even hard to find a research center similar to the IKZ anywhere in the world. Now, enhancing the competences in the field of lasers and crystals for optical applications makes it even more unique. I don't think that there is any other institute that has comparable knowledge and the equipment for the research that is done at the IKZ in my field.

Photo: E. Castellano © IKZ

Elena: In many laser groups, the PhD students are also growing the crystals, but they are not experts. This takes lots of time during the PhD. The situation here is much more beneficial. We have expert crystal growers that are responsible of this part of the work, so you can focus on your research.

Is it your first experience to establish a lab from the beginning? What are the specific challenges that you encountered?

Christian: I set up my labs when I started my group in Hamburg, but at that time I had, at least, an optical table. So I did not start from complete zero. Here, I started from a lab that was equipped for another purpose and had to be emptied and renovated, all the water piping had to be done from the beginning. This was kind of a new experience. Also, I had to buy an optical table, which is about 3x4 meters in dimensions and weights around two tons. It had to be brought here, on the third floor, by a crane. It was an experience, even for the people who delivered it.

Also, the new part for me was, coming from a university environment, to see how good the technical service here at the IKZ was prepared for this kind of job. There was a lot of support by, particularly, Jens Klose and his people. That was a good experience.

Now, of course, we are still struggling with the Berlin's tender regulations for buying expensive stuff, but, nevertheless, I think, we have gone a good way – we have a functioning lab with a lot of state-of-the-art equipment. That is the fun part of the research.

Elena: For me, it was a new experience to acquire equipment. It is very interesting because you get an idea about the price of the things you work with. You learn a lot about the devices you are using just because you have to check all the characteristics before you decide which one to buy. It is also good that you have a chance to build up everything according to your needs and your taste.

Was sind derzeit die wichtigsten Projekte im Zentrum und welche Ziele verfolgen sie?

Christian: *Am Anfang stand die Förderung des BMBF-Projekts EQuiLa, das es uns grundsätzlich ermöglichte, die Arbeit am ZLM zu beginnen. Die Themen dieses Projekts sind: erstens, Terbium-dotierte Materialien für sichtbare Laser; zweitens, Erbium-dotierte Sesquioxide für Laser im mittleren Infrarotbereich, die Kristalle werden mit dem optischen Zonenschmelzverfahren gezüchtet; und das dritte Thema bearbeiten wir in Zusammenarbeit mit dem FBH. Sie liefern InGaP-basierte rote Laserdioden, die zum Pumpen von Cr:LiCaF-Kristallen verwendet werden. Das ist bemerkenswert: Damit sind wir in Adlershof in der Lage, sowohl das aktive Verstärkungsmaterial züchten, als auch die Halbleiterpumpenquelle am FBH herstellen zu lassen und einen Laser-Demonstrator aufzubauen. Hiermit wird die gesamte Wertschöpfungskette eines Lasers abgedeckt. Ich denke, man findet keinen anderen Ort auf der Welt, an dem all dies in einem Umkreis von fünfhundert Metern möglich ist.*

In enger Zusammenarbeit mit Matthias Bickermann (Red: Abteilungsleiter Dielektrika & Wide Bandgap am IKZ) und Partnern aus der Industrie beteiligen wir uns auch am BMBF-Projekt IsoNova, wo wir Kaliumterbium-fluorid (KTF)-Kristalle für optische Isolatoranwendungen untersuchen.

Dann gibt es noch ein weiteres Projekt, in dem wir beratend tätig sind, um Empfehlungen für die Entwicklung von Lasern und aktiven Materialien im mittleren Infraroten Spektralbereich in Deutschland zu geben und Wachstumstechnologien zu finden, die es derzeit in Europa noch nicht gibt.

Darüber hinaus haben wir mehrere weitere Vorschläge für neue Projekte eingereicht, z.B. zu Ytterbium-dotierten Sesquioxid-Materialien.

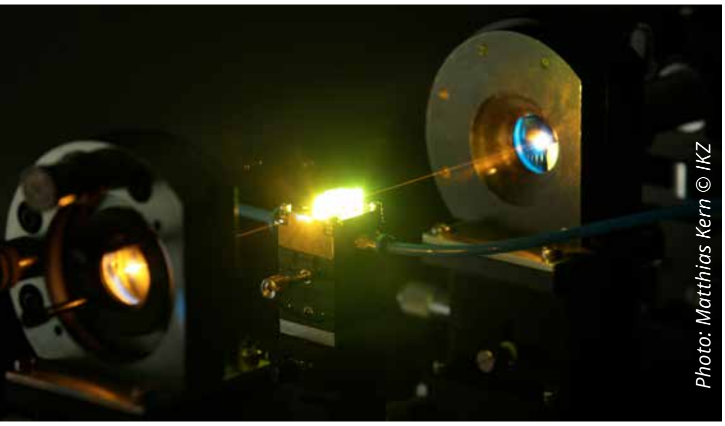


Photo: Matthias Kern © IKZ

Blue-pumped directly yellow emitting laser with a Tb:LLF crystal as an active material.

Blau gepumpter direkt gelb emittierender Laser mit einem Tb:LLF-Kristall als aktivem Material.

Habt ihr bisher schon interessante Ergebnisse erzielt?

Elena: *Wir untersuchen im Rahmen meiner Doktorarbeit Terbium-dotierte Lasermaterialien. Vor kurzem haben wir den bisher stärksten direkt gelb emittierenden Festkörperlaser aufgebaut. Die Ausgangsleistung ist etwa eine Größenordnung stärker als bei allen bisherigen Untersuchungen.*

Christian: *Und auch effizienter, was für die weitere Skalierung wichtig ist.*

Ein weiteres interessantes Ergebnis ist meiner Meinung nach die Sichtbarkeit, die wir mit diesem Institut bereits erreicht haben. Wir hatten bereits Gaststudenten aus Hamburg, Japan und Weißrussland. Darüber hinaus haben wir eine Marie-Curie-Studentin aus Großbritannien angeworben. Das zeugt von unserer internationalen Sichtbarkeit. Wir haben bereits aus der ganzen Welt Einladungen zu Vorträgen erhalten. Das zeigt mir deutlich, dass es einen Bedarf an dieser Art von Forschung gibt, wie wir sie hier durchführen.

Was sind Ihre Vorstellungen für die Zukunft des Zentrums für Lasermaterialien?

Christian: *Ich möchte das Zentrum als das führende Forschungsinstitut für Lasermaterialien in Deutschland, wenn nicht gar in Europa etablieren. Unser Ziel ist es, eine zentrale Anlaufstelle für alle Fragen zu Lasern, Verstärkungsmaterialien, spektroskopischen Eigenschaften oder unerwünschten Verunreinigungen in Kristallen zu sein. Wenn jemand ein Problem mit seinen Festkörperkristallen hat, möchte ich derjenige sein, der den Hörer abnimmt und dieser Person hilft.*

Neben unserer laufenden Forschung an sichtbaren Lasern und infraroten Lasern denke ich, dass in Zukunft der Spektralbereich des mittleren Infrarots jenseits von 3 µm ein wichtiges Forschungsthema sein wird. Für diese Anwendungen kommen auch nicht-oxidische, nicht-fluoridische Wirtsmaterialien mit kleiner Bandlücke in Frage, also Halbleitermaterialien oder Chalkogenide, aber auch andere Materialien.

Ein weiterer Punkt, den wir auch in Zukunft in unser Portfolio aufnehmen werden, sind nichtlineare Materialien. Diese können sowohl für die Frequenzverdopplung – hier meist Oxide und einige Fluoride – als auch für die parametrische Konversion von nah-infrarotem Licht in fernes oder mittleres Infrarot verwendet werden. Dies sind wiederum meist Halbleitermaterialien mit einer entsprechenden Transparenz im mittleren Infrarot-Spektralbereich. Das werden in Zukunft sehr heiße Themen sein, aber das ist eher eine langfristige Perspektive.

What are the main projects running in the Center now, and what are their goals?

Christian: Initially there was the funding of the BMBF project *EQuiLA*, which basically allowed us to start the work at the ZLM. The topics of this project are: first of all, terbium-doped materials for visible lasers; second, erbium-doped sesquioxide materials, grown by the optical-floating zone growth technique for mid-infrared lasers; and the third topic is in cooperation with the FBH. They deliver InGaP-based red laser diodes which are utilized to pump Cr:LiCaF crystals. That is a very interesting thing: then we are able to grow the active gain material, to fabricate the semiconductor pump source at the FBH, and to set up the laser demonstrator. This is the complete value chain of a laser. I think you cannot find any other place in the world where all this is possible within a range of five hundred meters.

We also participate in BMBF project *IsoNova* in close collaboration with Matthias Bickermann *(Ed: Department head of the Dielectrics & Wide Bandgap Materials at the IKZ)* and other industry partners, where we investigate potassium terbium fluoride (KTF) crystals for optical insulator applications.

Then, there is another project where we are more working as consultants to give hints towards the development of mid-infrared lasers in Germany and to find the growth technics, which currently do not exist in Europe.

Moreover, we have submitted several further proposals for new projects, e.g., on ytterbium-doped sesquioxide materials.

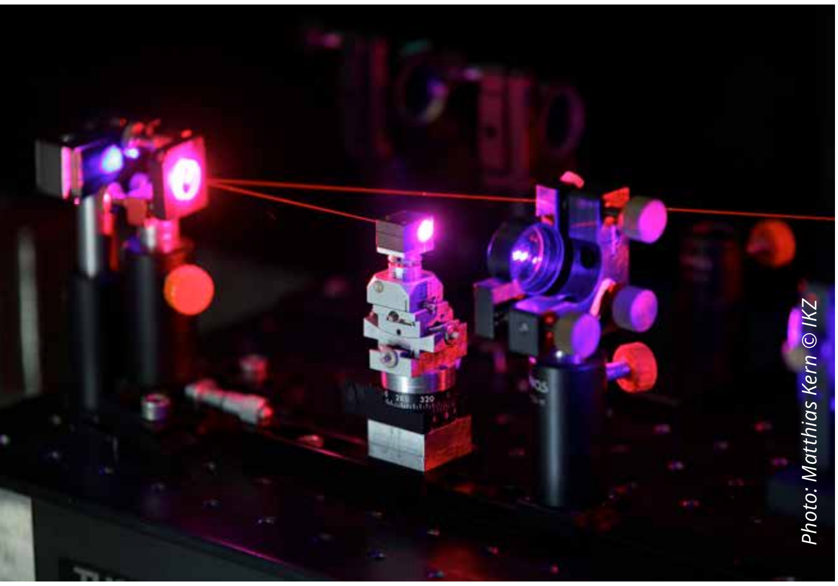


Photo: Matthias Kern © IKZ

Diode pumped Pr:YLF laser.
Diodengepumpter Pr:YLF-Laser.

Did you come up with some interesting results so far?

Elena: We are developing terbium-doped laser materials in the frame of my PhD. Recently, we got the most powerful direct yellow emission from a solid-state laser up to now, which is about one order of magnitude more powerful than in any previous research.

Christian: And also more efficient which is important when it comes to further scaling.

Another interesting result, in my opinion, is the visibility that we have already generated with this institute. We already had guest students from places like Hamburg, Japan, and Belarus. Besides, we attracted a Marie-Curie student from Great Britain. These are signs of international visibility. We have already received many invited talks to places all over the world. This clearly shows me there is a need for this kind of research that we are doing here.

Elena: We also have many international collaborations with France, Italy, Japan, US.

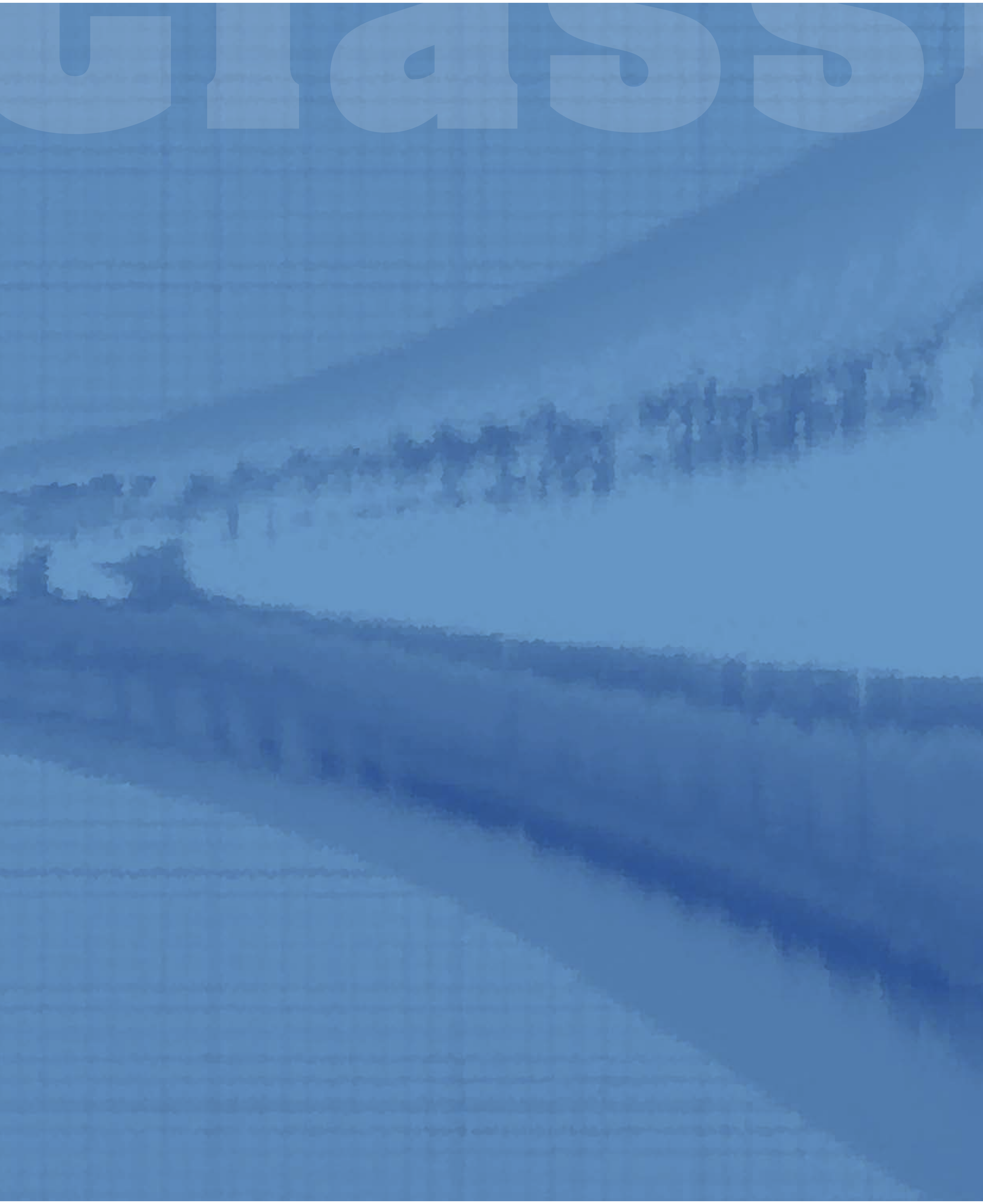
What are your visions for the future of the Center for Laser Materials?

Christian: I want to establish this center as the leading research institute for laser materials in Germany, if not in Europe. We target to be a one-stop-agency for all questions regarding lasers, gain materials, spectroscopic properties, or unwanted impurities in crystals. If someone has a problem with his solid-state crystals, I want to be the one who picks up the phone and helps this person.

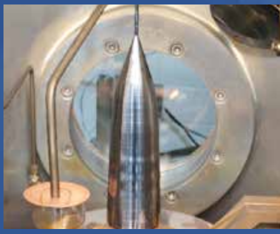
Besides our ongoing research on visible lasers and mid-infrared lasers, I think, in future the mid-infrared spectral range will be a hot topic. That will also cover non-oxide, non-fluoride host materials that will have to be utilized, saying, semiconductor host materials, chalcogenides might be a topic, but also other materials.

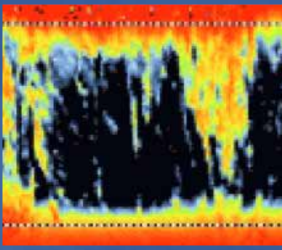
Another thing that we also consider to take up into our portfolio in the future is nonlinear materials. These are materials that could be used either for frequency-doubling – mostly oxide and some fluoride materials – but also materials to convert near-infrared light into far- or mid-infrared light, which then again are mostly semiconductor materials that allow transparency in the mid-infrared spectral range. These topics will be very hot in the future, but that's more on a long-term scale.

Klassische Halbleiter



Classical Semiconductors





Klassische Halbleiter

Acting head of department: Dr. Frank M. Kießling

Die Abteilung betreibt die Grundlagen- und angewandte Forschung im Bereich der Kristallzüchtung von Volumenkristallen aus der Schmelze der elementaren Halbleiter – Silizium (Si), Germanium (Ge) und deren Mischkristalle, sowie der klassischen III-V-Verbindungshalbleitermaterialien.

Die Abteilung arbeitet in engem Kontakt mit Industriepartnern und entwickelt Wachstumsprozesse bis hin zum industriellen Maßstab. Darüber hinaus ist sie auch in der Grundlagenforschung tätig, indem sie sich mit Fragen wie der Entstehung und Kontrolle von Defekten in Kristallen beschäftigt.

Als wesentliche Züchtungstechniken werden in der Forschung das Floating Zone- (FZ), das Czochralski- (CZ) und das Vertical Gradient Freeze-Verfahren (VGF) eingesetzt. Einige Züchtungsanlagen sind mit den patentierten KRISTMAG® *Heizmagnetmodulen ausgerüstet, um die Schmelzkonvektion während des Wachstumsprozesses durch das Anlegen von Wandermagnetfeldern effektiv zu steuern.*

Aktuelle Arbeitsgruppen in dieser Abteilung sind:
- *Silizium und Germanium*
- *Multikristallines Silizium*
- *Galliumarsenid*

Classical Semiconductors

The department carries out the fundamental and applied research in the bulk crystal growth from the melt of the elementary semiconductors – silicon (Si), germanium (Ge) and their solid solutions, as well as classical III-V compound semiconductor materials.

The department works in close contact with industrial partners and develops growth processes up to industrial scale. Apart from that, it is also active in fundamental research by tackling the questions like the formation and control of defects in crystals.

Floating Zone (FZ), Czochralski (CZ) and Vertical Gradient Freeze (VGF) are the principal growth techniques used in research. Patented KRIST*MAG*® heater-magnet-modules are embedded into some growth systems to provide effective control of the melt convection during the growth process by applying traveling magnetic fields.

Current working groups in this department are:

- **Silicon and Germanium**
- **Multicrystalline Silicon**
- **Gallium Arsenide**

Classical Semiconductors:
Silicon & Germanium

Head Dr. Nikolay Abrosimov
Team M. Czupalla, Dr. K. Dadzis, J. Fischer, B. Faraji-Tajrishi, O. Gybin, B. Hallmann-Seifert, Dr. J. Janicskó-Csáthy, S. Kayser, L. Lehmann, Dr. A. Lüdke, Dr. R. Menzel, Dr. W. Miller, K. Reinhold, M. Renner, Dr. H. Riemann, Dr. H.-J. Rost, T. Turschner, S. Weiß

Überblick

Silizium (Si) und Germanium (Ge) zählen zu den Halbleitermaterialien mit höchster Anwendungsbreite in Schlüsselindustrien wie zum Beispiel der Mikro- und Leistungselektronik, der Photovoltaik oder der Photonik. Die Kompetenzen der Themengruppe Silizium & Germanium liegen in der Züchtung dieser Elementkristalle und deren Si_xGe_{1-x} *Mischungen. Für die Züchtung von Si wird am IKZ vor allem das tiegelfreie Floating-Zone (FZ) Verfahren angewandt, welches die Herstellung von perfekten Einkristallen mit extrem hoher Reinheit erlaubt. Dieses Alleinstellungsmerkmal des FZ Verfahrens ermöglicht auch Anwendungen in der Grundlagenforschung oder zum Beispiel in der Metrologie.*

Unter Leitung der Physikalisch-Technischen Bundesanstalt (PTB) wurde das international vielbeachtete „Avogadro“ Projekt unter dem Namen „Kilogramm“-Projekt fortgeführt. Ziel des Projekts ist eine genauere Bestimmung der Avogadro-Konstante, die die Anzahl der Atome in einem Mol angibt und für eine neue Definition der Maßeinheit Kilogramm verwendet werden kann. Aufgabe des IKZ war die FZ-Züchtung von versetzungsfreien, isotopenreinen ^{28}*Si-Kristallen mit extremer Reinheit. Aus jedem gezüchtetem Kristall werden zwei Kugeln präpariert und deren Volumen bestimmt. Aus der Masse der Kugel und dessen Volumen kann unter Berücksichtigung von Atommasse und Gitterparameter die Avogadro-Konstante ermittelt werden. Im Berichtszeitraum wurden zwei neuen Chargen für isotopenreine* ^{28}*Si Kristalle fertiggestellt.*

Im Jahr 2017 begann das BMBF-Projekt mit den am GERDA (GERmanium Detector Array) Experiment beteiligten Instituten. Im GERDA Experiment wird nach dem neutrinolosen Doppel-Betazerfall in ^{76}*Ge gesucht. Das Projekt ist eingebettet in die internationalen Aktivitäten zur Entdeckung der Natur der Neutrinos. Obwohl diese nach Photonen die zweithäufigste Art von Teilchen im Weltraum sind, sind ihre Eigenschaften noch weitgehend unbekannt. Die Aufgabe des IKZ besteht in der Züchtung von hochreinen Ge Kristallen mit definierter Versetzungsdichte, welche für Detektoren in den Experimenten eingesetzt werden können. Die Herausforderung besteht darin, das Germanium bis zu einem Niveau unterhalb 1 ppt zu reinigen.*

Die wissenschaftlich-technische Weiterentwicklung der Züchtungsmethoden blieb auch in 2017 ein Schwerpunkt der Gruppe. Im von der Leibniz-Gemeinschaft im Rahmen des im Leibniz-Wettbewerb geförderten Vorhabens SiGrEt (Silizium-Granulat Eigentiegelverfahren) wird ein neuartiges Züchtungsverfahren entwickelt. Mit dem Konzept könnten die Vorteile der industriell etablierten Methoden zur Herstellung von monokristallinem Silizium vereint und die Nachteile vermieden werden. Ziel des Projektes ist die Demonstration eines stabilen Züchtungsprozesses für Kristalle mit großem, industrierelevantem Durchmesser und hoher Reinheit. Im Jahr 2017 konnten wesentliche Meilensteine erreicht werden. So konnten mit dem entwickelten Versuchsaufbau reproduzierbar Kristalle mit einem Durchmesser bis 3 Zoll und einem sehr niedrigem Sauerstoffgehalt ($<10^{16}$ *a/cm*^{3}*) auf dem Niveau des FZ-Si Materials gezüchtet werden. Erstmals gelang die Züchtung von einkristallinem Material.*

Die numerische Modellierung war weiterhin von großer Bedeutung für die Weiterentwicklung unserer Kristallzüchtungsprozesse und Anlagen, wurde aber auch von unseren Industriepartnern nachgefragt. Im Auftrag des Anlagenbauers PVA TePla CGS wurden dreidimensionale Berechnungen des elektromagnetischen Feldes beim induktiv beheizten FZ Verfahren für Silizium Kristalle großen Durchmessers durchgeführt.

In Rahmen des BMBF Projektes InTerFEL (Zeitaufgelöste und nichtlineare Infrarot- und Terahertz-Spektroskopie mit Freien Elektronen Lasern) haben wir ein neues Verfahren zur Phosphordotierung von Germaniumkristallen mit der sogenannten „Mini-Czochralski“ Züchtungsmethode entwickelt.

Classical Semiconductors: **Silicon & Germanium**

Overview

Silicon (Si) and Germanium (Ge) are semiconductor materials with wide application in micro- and power-electronics, photonics or photovoltaics. The group Silicon & Germanium is active in the growth of these element crystals and its Si_xGe_{1-x} mixtures. For the growth of Si, primarily the crucible-free Float Zone (FZ) method is used, which allows production of perfect dislocation-free single crystals with ultra-high purity. This unique feature of the FZ method also leads to the application in fundamental research or, e.g., in metrology.

Headed by the Physikalisch-technische Bundesanstalt (PTB) the internationally renowned "Avogadro" project continued under the name "Kilogramm" . The aim of the project is to determine more precisely the Avogadro constant, i.e., the number of atoms in one mole, which may serve for a new natural definition of the kilogram mass unit. The task of IKZ was the FZ growth of dislocation-free monoisotopic ^{28}Si crystals with extreme purity. From the measured volume, mass, and lattice parameter of a Si sphere prepared from the grown material, it is possible to deduce the number of atoms and determine the Avogadro constant. In the reporting period, two new charges for monoisotopic ^{28}Si crystals were completed.

2017 marks the starting of the joint BMBF project with the institute participating in the GERDA (GERmanium Detector Array) experiment, which is dedicated to the search of neutrinoless double beta decay in ^{76}Ge. The project is embedded in the international activities to discover the nature of neutrinos. Although these are after photons the second most common type of particles in space, their properties are still largely unknown. Their understanding could provide fundamental insights into the development of the universe and in the field of particle physics. The task of IKZ in this project is to grow high-purity Ge crystals with defined dislocation density that can be used for detectors production in the experiments. The challenge is to purify the Germanium to the level below 1 ppt.

The scientific and technical development of crystal growth methods remained the main focus of the group's activities. In the project SiGrEt (Silicon growth from a silicon granulate crucible), funded by the Leibniz Association in the frame of the Leibniz Competition, the group develops a novel growth method. The growth concept allows to combine the advantages and avoid the disadvantages of the industrial established methods for the production of monocrystalline Si. The project aims to demonstrate a stable growth process for crystals with industrial relevant dimensions and high purity. In 2017, we achieved reproducible growth of single crystals with a diameter of up to 3 inches and a low oxygen content ($<10^{16}$ a/cm^3) at the level of FZ-Si material.

Numerical modeling was essential for the further development of our growth processes and equipment but was also in high demand by our industry partners. For the equipment manufacturer PVA TePla CGS, we conducted three-dimensional calculations of the electromagnetic field distribution during FZ growth of large-diameter Si crystals.

In the frame of the BMBF project InTerFEL (Time-resolved infrared and terahertz spectroscopy of carrier dynamics in a semiconductor with free electron lasers), we developed a new method for the phosphorus-doping of Ge crystals using the so-called "mini-Czochralski" growth technique.

Results

Development of high-purity germanium crystal growth

As part of our contribution to the joint BMBF GERDA project, more than 30 Ge crystals were grown in H_2 atmosphere in 2017. All the grown crystals had a diameter of about 2" with a mass of about 1.5 kg. They were fully characterized with Hall-effect measurement, Photo-thermal Ionization Spectroscopy (PTIS), and the dislocation density was measured with the Etch Pit Density (EPD) method. Based on the PTIS spectra, aluminium, boron, phosphorus and gallium were identified as the dominant impurities with concentrations varying between 10^{10} to 10^{12} atom/cm^3 in the analyzed samples. In separate experiments, we reduced the concentration of individual impurities, but the main target is to reduce net impurity concentration down to 10^{10} atom/cm^3.

The temperature field of the Czochralski furnace was further optimized using numerical modeling. Adding heat insulation around the crucible as suggested by the simulation helped to achieve the target values for dislocation densities. One of the crystals had the dislocation density of about 5000 cm^2, which is well below the required maximum of 10,000 cm^{-2} for p-type crystals. The grown crystals had a diameter of about 2". To reach the required 3" crystal diameter, we performed simulations for a new setup inside the Czochralski furnace with 6" crucible diameter. We plan to complete the installation of this setup in 2018.

In 2017, the group successfully set up its own zone-refining furnace and was able to routinely purify germanium in pure H_2 atmosphere. With this step, we completed setting up the production chain for high purity Ge at IKZ. Several crystals were already grown from the purified germanium, using the optimized growth setup.

Fig. 1
Zone refined Ge ingots.

Fig. 2
2 inches Ge crystal.

To provide a long term perspective to the “High Purity Germanium” project at IKZ, we are planning to continue our GERDA activities as a member of the LEGEND collaboration. This collaboration has been established in 2017 to build an experiment to search for the neutrinoless double beta decay. This experiment will give insight in the nature of neutrinos and could prove whether they are their own antiparticles. If so, this could provide an explanation for the surplus of matter in the universe. The first phase of the experiment is planned to start in 2020 and will require 200 kg of detectors made of germanium enriched with the ^{76}Ge isotope. Later, a total of 1 t of germanium detectors will be needed, generating high demand for high purity Ge crystals.

Development of the Si growth from Si granulate crucible (SiGrEt project)

Substantial progress was achieved in the project SiGrEt, in which a novel crystal growth technique for the production of Si single crystals is developed. In contrast to the Czochralski method, the crystal is not contaminated by a crucible material, and the used quartz container does not degrade, as it is not in contact with the hot Si melt. The silicon self-crucible contains the inductively heated (f = 2 MHz) melt pool that stabilizes in a bed of Si granules. The crystal is pulled with a rate of 12 mm/min upwards through a central hole in the inductor. High-purity Si granules are used as feed material, and are available at fraction of the cost of the expensive Siemens feed rods needed for FZ technique. As in the FZ method, a homogeneous axial dopant distribution in the crystal is achievable. A goal of the project is to demonstrate a stable growth process for crystals with industrial relevant diameter of 4 inches.

Growth parameters for scale-up of the crystal diameter were obtained using a global 2D transient numerical model of the SiGrEt process. The finite element model allowed time-dependent simulation of the electromagnetic and temperature field, including phase boundaries, for all process steps from generation of the melt pool to necking and pulling of the crystal. For calculation of the self-crucible shape, a phase field method, considering the three phases – Si solid, Si liquid, and Si granular bed, showed to be applicable with good agreement to the experimental results. The shape of the crystal was calculated using a triple-point model [1]. As shown in Fig. 3, an almost flat crystallization interface was obtained for crystals with diameter of 3 inches and pull rate of 2 mm/min, which is in agreement with the experimental findings. For growth of crystals with diameter of 4 inches, a convex interface shape was predicted, due to the diminishing heat introduction to the center of the melt pool, as a consequence of the larger inductor hole.

For the first time, we have grown a single crystal using the SiGrEt method (see Fig. 4). The growth of a crystal without grain boundaries was challenging for several reasons. For the successful seeding procedure, we used a special shaped starting-susceptor to heat the seed as well as the center of the melt pool. The main difficulty was to set a time-stable surface temperature near Si melting point for generation of a proper melt meniscus during growth of a Dash neck. Two pyrometers were used to simultaneously measure the temperature of the starting-susceptor and the melt surface.

We observed that the thermal conditions change drastically while the starting-susceptor is pulled upwards out of the electromagnetic field generated by the inductor. The correct determination of values for heater power (32 kW), seed rotation (8 u/min) and pull rate (8-10 mm/min) was essential to transfer the seeds <100> orientation to the crystal. To preserve the single-crystalline growth, any strong temperature fluctuations and contact of solid particles with the crystallization interface must be avoided. Such particles may be umolten Si granules but also SiO that deposited at the cold lower inductor surface and they can fell down to the melt pool. The melting of Si granules was improved with the inductor shape and optimal value for crucible rotation, inducing a favorable melt flow directed away from the triple point line. The formation of SiO during melting was significantly reduced by preheating the Si granules for 3h in high vacuum, to thoroughly evaporate H_2O from the large surface area of the granules.

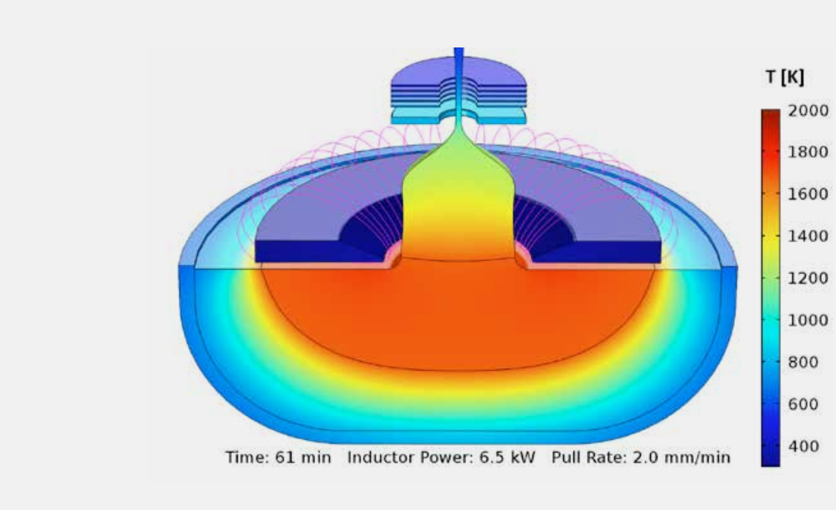


Fig. 3
Calculated temperature field, phase boundaries and crystal shape during growth of a 3 inches crystal using the new SiGrEt method.

Fig. 4
Si single crystal grown using the developed SiGrEt setup.

The experimental setup was equipped with a system for the in-situ measurement of the crystal diameter. The knowledge of the actual crystal diameter during growth is important for the adjustment of the replenishment rate of fresh granules to the melt pool. If the mass balance is not preserved, the consequent rise or fall of the filling level of the melt pool deteriorates the process stability. The developed system directly detects a part of the triple-point line with high-resolution cameras and uses edge and object detection algorithms to calculate the crystal diameter.

Phosphorus-doping of germanium crystals

The growth of phosphorus-doped Ge crystals was a challenge of the BMBF project InTerFEL on infrared and terahertz spectroscopy using a Free Electron Laser (FEL). Here, the development of a new doping method from the gas phase was required. The problem with the phosphorus doping ($k_0 = 0.08$) of Ge is the lack of stable dopants. Elemental phosphorus cannot be used due the low chemical stability of phosphorus at the melting temperature of Ge. Therefore, the gaseous chemical compound phosphane (PH_3) was chosen as a phosphorus source. Phosphane is typically used for the growth of phosphorus doped Si crystals by the FZ method. The phosphorus-doped Ge crystals were grown in a FZ system using the so called Mini-Czochralski method [2]. A peculiarity of this method is the use of inductive heating with a frequency of f = 3 MHz. This allows stable growth conditions, even in the case of very little starting material. The process begins with melting of Ge in a quartz crucible. The doping starts by blowing phosphane to melt surface using a special nozzle (see Fig. 5). After the gas contacts with the melt surface, which has at least the melting temperature of Ge (938 °C), it dissolves into the elements hydrogen and phosphorus. The amount of phosphorus introduced into the melt in this way depends on the total time of exposure. After the doping is finished, the nozzle is removed from the melt and the growth by Czochralski technique starts. In this manner, we pulled phosphorus-doped Ge crystals with a charge carrier density of 10^{14} cm^{-3} to 10^{16} cm^{-3}.

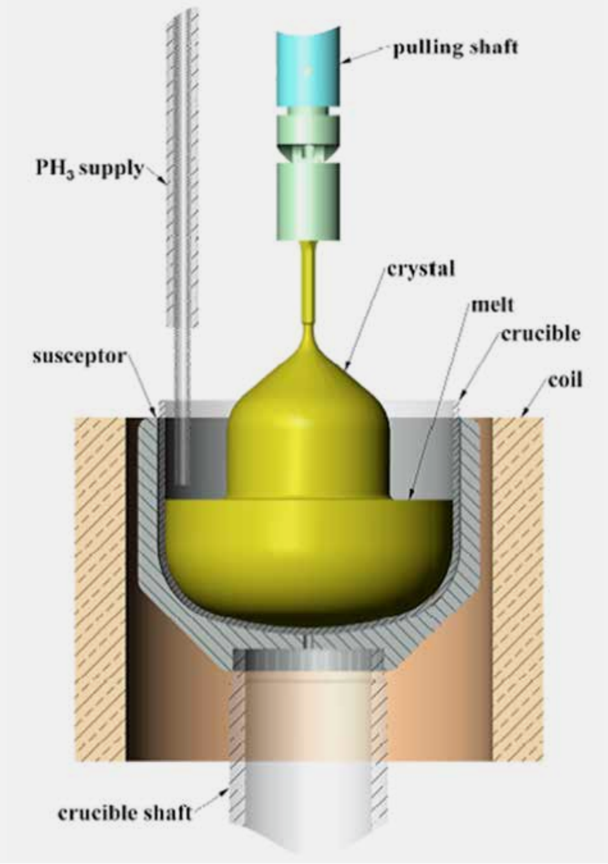


Fig. 5

Scheme of the growth of phosphorus-doped Ge crystals using the mini-Czochralski method.

Further development of Lateral-Photovoltage-Scanning method

The detection of doping inhomogeneities in grown semiconductors such as Si, Ge and Si_xGe_{1-x} is the main idea of the Lateral-Photovoltage-Scanning method (LPS). The LPS method allows to determine the shape and deflection of the crystallization interface. In contrast to destructive techniques, such as defect etching and Secondary Ion Mass Spectroscopy (SIMS), the LPS method offers a non-destructive option for wafer-size samples. The LPS method works as follows: a modulated laser excites locally free charge carriers (electrons and holes), which will diffuse through the semiconductor. If there is a local gradient in the charge carrier density, a directed drift process of these charge carriers takes place, which will lead to a local dipole. The resulting electrical potential is detected with respect to the modulation frequency at the ohmic rim contacts.

The theory about the photovoltaic phenomenon in semiconductors, on which LPS-measurements rely on, was developed by Tauc [3] in 1955. He assumed a one-dimensional sample with constant charge carrier mobility, constant charge carrier life time and constant illumination. The actual setup [4] oppose this assumptions using a spot-like laser focus. Also, multicrystalline silicon sample were measured, which heavily infringe Tauc's assumptions. Hence, a theory is needed, which does not rely on this assumptions. Therefore, we used a finite-volume approach in three dimensions using COMSOL Multiphysics [5]. We compared this simulation with real measurements and Tauc's theory and observed the good agreement (Fig. 6). It proves that a finite-volume approach is suitable to describe the LPS-method. First of all, this opens opportunities to describe three-dimensionally structured samples, which are common nowadays due to ion implantation. Also this allows to discuss the influence of grain boundaries or different recombination mechanisms in a semiconductor sample. In addition, the different wavelength of the lasers are now described well by a penetration depth in the sample. So the finite-volume approach is heavily in favor to the theory of Tauc.

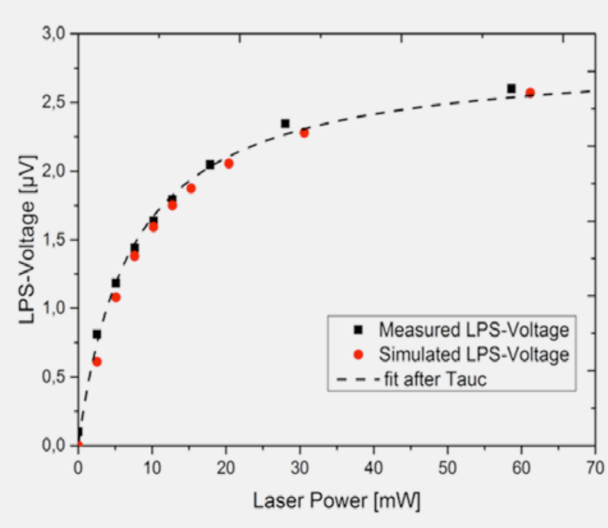


Fig. 6

Dependency of the measured LPS-voltage correlated to the power of the used laser. Also shown is the theoretical dependency introduced by Tauc and the computational simulation results using a finite volume approach. All three are in good agreement.

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Classical Semiconductors: **Multicrystalline Silicon**

Head Dr. Frank M. Kießling
Team I. Buchovska, Dr. N. Dropka

Überblick

Solarstrom ist eine saubere Alternative zu Strom aus fossilen Brennstoffen, da er ohne Luft- und Wasserverschmutzung, ohne Schadstoffemission, die zur Erderwärmung beiträgt und ohne Gefährdung der öffentlichen Gesundheit erzeugt wird. Entsprechend ist die Photovoltaik ein immer noch stetig wachsender Markt. Allein in Deutschland stieg die PV-Leistung in 10 Jahren von 0,5 % auf 7,5 % des gesamten Nettostromverbrauchs. Verschiedene berechnete Szenarien prognostizieren ein jährliches, weltweites Marktvolumen zwischen 40 GW und 60 GW installierte Leistung bis 2025. Die Si-Wafer-basierte PV-Technologie nimmt davon etwa 95% des Weltmarktes ein und der Anteil der multikristallinen Technologie wird für die nächsten 15 Jahre auf mindestens 40% der Gesamtproduktion geschätzt. Das bedeutet, dass die weltweite Nachfrage nach multikristallinem Material mindestens 15 GW/Jahr betragen wird. Leider findet zur Zeit die industrielle Kristallisation, welche in der Wertschöpfungskette zum Modul am Anfang steht, überwiegend im außereuropäischen Raum statt, so dass diese Forschung- und Entwicklungsaufgaben nur noch bedingt in Europa nachgefragt sind.

Dieses enorme Wachstum des multikristallinen Si-Marktes zusammen mit einer bemerkenswerten Senkung der PV-Leistungskosten erfordert eine verbesserte Technologie zur Herstellung von billigem, multikristallinem Si-Material bei gleichzeitiger Verbesserung seiner Qualität. Weltweit werden kontinuierlich neue Solarzellen-Designs und Produktionstechnologien entwickelt. Diese bieten die Möglichkeit, den Zellwirkungsgrad auf bis zu 25% relativ zu steigern. Voraussetzung dafür ist kristallines Material von höchster Qualität (bessere Struktur, höhere Reinheit, spezielle Eigenschaften usw.), das noch nicht auf dem Markt verfügbar ist. In der Vergangenheit hat unsere Gruppe Verfahren für drei Typen von p-Typ Siliziummaterial – multikristallines (mc), hochleistungsfähiges mc (HPmc) und quasi-mono (QM) Silizium – durch gerichtete Erstarrung unter spezieller Verwendung von Magnetfeldern entwickelt. Neuesten Berichten von weltweit führenden PV-Forschungsinstituten zufolge, fokussieren diese ihr Interesse insbesondere auf die Entwicklung von hocheffizienten Solarzellen auf Basis vom Typ Quasi-Mono, HP-mc und n-Typ multikristallinen Materialien. Für n-Typ Siliziummaterial waren bisher nur Zellkonzepte für monokristalline Wafer bekannt. In letzter Zeit wurden aber auch Solarzellenkonzepte für die deutlich preiswerteren n-Typ mc-Si Wafer entwickelt. Die Gruppe hat bereits mit der Entwicklung von Wachstumsprozessen für n-Typ Siliziummaterial begonnen. N-dotiertes Silizium hat höhere Standzeiten und damit ein höheres Wirkungsgradpotenzial als p-dotiertes Silizium. Forschungs- und Entwicklungsbedarf bestehen bei diesem Materialtyp hinsichtlich einer homogenen Verteilung geeigneter Dotierstoffe bei gleichzeitiger Reduktion rekombinationsaktiver Kristalldefekte.

Leider ist die Produktions- und Forschungssituation im PV-Bereich in Europa stark rückläufig. Dennoch hat die multikristalline Siliziumgruppe ihre Grundlagen- und Projektforschung erfolgreich fortgesetzt. Insbesondere der Transport von Phosphor für die n-Typ-Dotierung in der Schmelze zur fest-flüssig-Phasengrenze hin und von dieser weg stand im Mittelpunkt der aktuellen Forschungsaktivitäten. Diese Transportmechanismen werden dabei durch dynamische Magnetfelder beeinflusst, welche über das inzwischen weltweit bekannte KRISTMAG®-Heizermagnetmodul erzeugt werden. Die Prozessentwicklungen wurden vollständig von der numerischen Modellierung begleitet. Die Ergebnisse wurden in enger Zusammenarbeit mit den IKZ-Gruppen „Kristallbearbeitung“ und „Physikalische Charakterisierung“ erzielt. Auch hat sich der Lebensdauermessplatz (MDP-Methode) etabliert und es wurden Proben von am Standort Adlershof ansässigen Instituten gemessen.

Overview

Solar electricity is a clean alternative to electricity from fossil fuels and provides a positive environmental impact, with no air and water pollution, no global warming pollution and no threats to the public health. Photovoltaics market grows rapidly – only in Germany PV-generated power increased in 10 years from 0.5 % to 7.5% of the total net electricity consumption. Different calculated scenarios predict annual worldwide market volume between 40 GW and 60 GW installed power by 2025. Si-wafer based PV technology takes about 95% of the world market, and the share of multi-crystalline technology is expected to be at least 40% of total production for the next 15 years, which means that the world demand for the multi-crystalline material will be at least 15 GW/year. Unfortunately, industrial crystallization, which is at the beginning of the module value chain, is currently taking place predominantly outside Europe, so that these research and development tasks are only in limited demand in Europe.

The tremendous growth of the multi-crystalline Si market together with its remarkable decrease of PV-power cost induce the need of improved technology for production of cheap multi-crystalline Si material while improving its quality. New solar cells designs and production technologies are continuously developed worldwide providing the opportunity to increase the cell efficiency by up to 25%. However, crystalline material of the highest quality is required (better structure, higher purity, exceptional properties, etc.), which is not yet available on the market. In the past, our group has developed processes for three grades of p-type silicon material - multi-crystalline (mc), high-performance mc (HPmc) and quasi-mono (QM) silicon - using directional solidification under the specifically applied magnetic fields. In particular, latest reports from world-leading PV research institutions show that they focus their interest on the development of high-efficiency solar cells based on quasi-mono, HP-mc, and n-type multi-crystalline materials. Until recently, cell concepts for n-type silicon material have been developed only for monocrystalline wafers, but solar cells concepts for less expensive n-type mc-Si wafers start to emerge. Our group already started to develop growth processes for n-type silicon material. N-doped silicon has higher lifetimes and, accordingly, a higher efficiency potential than p-doped silicon. Research and development of this type of material for homogeneous distribution of suitable dopants with a simultaneous reduction of recombination-active crystal defects are highly relevant.

Unfortunately, the production and research situation in the PV sector in Europe is declining sharply. Nevertheless, the multi-crystalline silicon group has successfully continued basic and project research. In particular, the transport of phosphorus for n-type doping in the melt to and from the solid-liquid phase boundary was the focus of current research activities. These transport mechanisms are influenced by dynamic magnetic fields generated by the KRIST*MAG*® heater magnet module, which is now known worldwide. The process developments were fully accompanied by numerical modeling. The results were achieved in close cooperation with the IKZ groups "Crystal Machining" and "Physical Characterization". The lifetime measuring station (MDP method) has also been established, and samples from institutes located at the Adlershof site have been measured.

Results

Towards graphite-free hot zone for directional solidification of silicon

In the solar cells production, it is still a crucial challenge to decrease impurities in directionally solidified Si ingots, especially, carbide (SiC), nitride (Si_3N_4) and transition metals (e.g. Al, Ca, Cr, Cu and Fe) to the degree necessary to render the long lifetimes and prevent cracks by ingot slicing. Particularly strong contamination comes from the graphite parts in the hot zone.

In the frame of the "CleanSi" project in cooperation with FCT Ingenieurkeramik GmbH, we were looking for massive ceramic materials which can replace standard graphite parts: crucible cover and support. The following eight ceramics were considered: titanium carbide (TiC), sintered silicon carbide (SSiC), recrystallized silicon carbide (RSiC), nitride bonded silicon carbide (NSiC), tantalum carbide (TaC), tantalum nitride (TaN), vanadium carbide (VC) and tungsten carbide (WC). Criteria for preliminary comparing assessments were: the chemical stability at high working temperatures in oxidative ambience, the level of metallic impurities, the health risks, the mechanical strength/durability and, finally, the price.

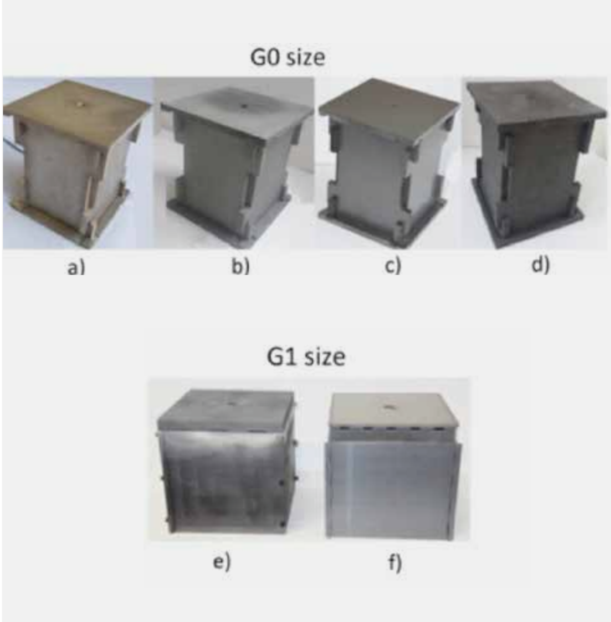

Fig. 1
New design of crucible support and cover at two scales G0 and G1 made of various materials: a)TiC, b) RSiC, c) SSiC, d) graphite, e) IKZ benchmark and f) TiC.

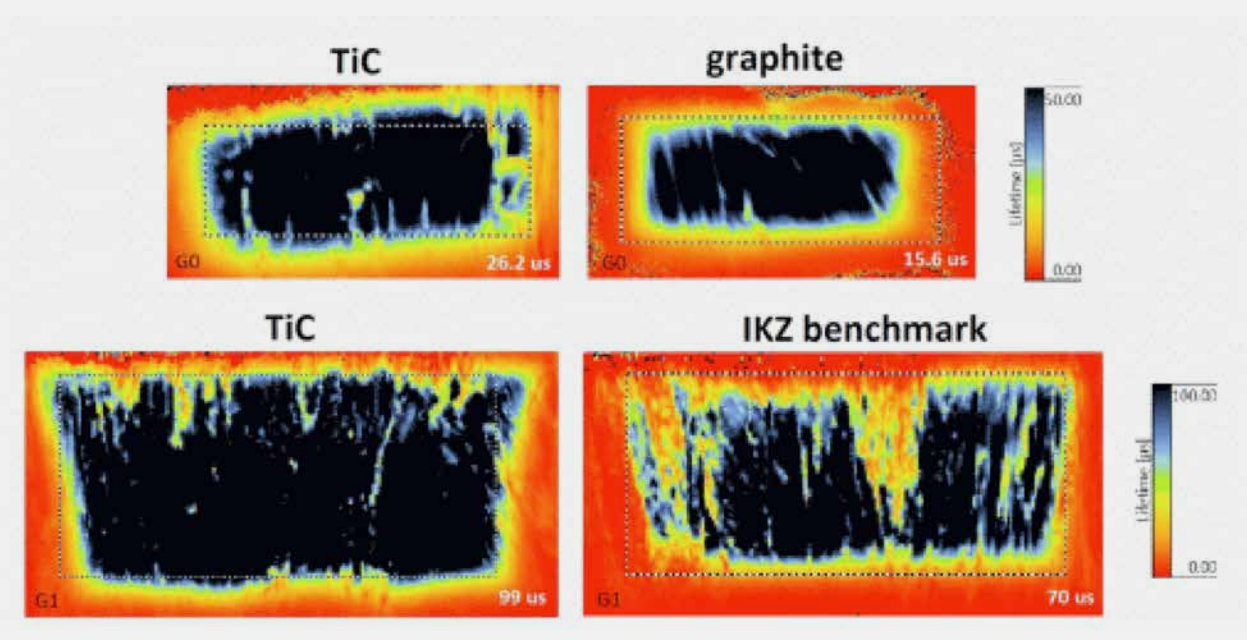

Fig. 2
MDP maps of minority carrier lifetimes in silicon ingots grown in graphite and ceramic setups of G0 and G1 size. Average lifetime values were determined for a central rectangle.

For three of the most feasible ceramics, TiC, RSiC and SSiC, we performed experimental tests. For this purpose, we adjusted the design of the crucible support/cover (Fig. 1) and the growth process to the material properties of each ceramic. The experiments were performed for phosphorus n-doped silicon of G0 and G1 size (0.9 and 14 kg). The Si ingots grown in the new ceramic parts were compared with ingots from the standard graphite design. The ingots were investigated with respect to O- and C-content, metal impurities, resistivity and minority carrier lifetime.

The first very encouraging results showed the superior performance of TiC ceramics in all aspects, particularly due to the high minority carrier lifetime with narrow red zones (Fig. 2, [1]).

Tailoring resistivity profile of phosphorus doped multi-crystalline silicon ingots by means of travelling magnetic field

Modern development of solar cell technology is focused on high-efficiency devices of novel concepts based on n-type crystalline Si material. However, due to high segregation of phosphorus in silicon, crystallized ingots have significant variation of resistivity along their height. This effect brings additional difficulties for cell manufacturing, as the industrial process is adjusted for significantly narrower resistivity variation, which results in lower production yield, and thus higher costs. Therefore, there is a strong need for specific solutions that will help to obtain relatively homogeneous axial resistivity profile along the height of phosphorus doped silicon ingots.

In this research, traveling magnetic field (TMF) was applied to silicon material during directional solidification (DS) in order to enhance or reduce mixing of silicon melt, and thus, to change fluid dynamics and dopant profile along the ingot. As the partial vapor pressure of phosphorus at concentrations standard for PV silicon is significantly higher than that of silicon at the silicon melting point, it volatilizes above Si-melt and can be removed from the system by ambient gas flow [2]. In preliminary work, we have already proved that enhanced melt stirring created by TMF can significantly increase evaporation of phosphorus from the melt surface, and thus noticeably change its distribution in the crystallized ingot.

Additionally, intentional influence on melt convection and impurity transport during crystallization process can alter dopant transport from melt volume to solid-liquid interface and change the effective segregation coefficient of phosphorus [3]. Consequently, it was expected that application of TMF of various strength at different process steps during DS might be an effective way to influence and tailor resistivity distribution along the ingot height in order to obtain multi-crystalline Si-material with homogeneous quality.

A row of experimental G0 ingots (0.9 kg) doped with an identical quantity of phosphorus were grown under the influence of TMF with variable strength. Enhanced mixing was performed on different stages of DS process (melting, melt homogenization, crystallization) and was kept either constant or intensified as the ingot was grown (Fig. 3). The resistivity of G0 ingots was measured by 4-probe method, and the values were compared one with another (Fig. 4). Since all the parameters including charge composition, crucibles and their coating, ambient gas pressure, gas flow, temperature and heat profiles were identical, the difference in resistivity profiles is attributed to the difference in strength and intensification of Si-melt stirring. The experimental data indicate that it is possible to tailor resistivity profile along at least 70% of G0 ingot length by using TMF of variable strength on different stages of DS. Consequently, this approach is a promising technique for growing phosphorus doped multi-crystalline ingots with more homogenous quality. The experimental work for scaling-up the results to larger ingots is in progress.

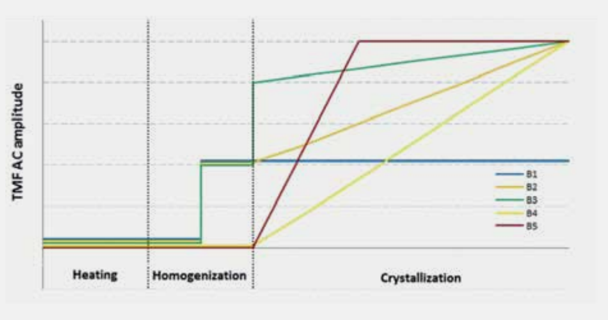

Fig. 3
Schematic profile of the intensity of Si-melt stirring with TMF on different stages of DS process (melting, melt homogenization and crystallization).

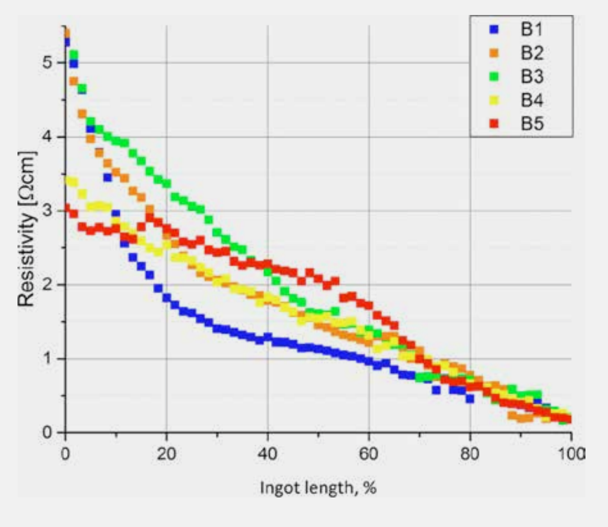

Fig. 4
Resistivity profiles measured along the central axis of 5 experimental ingots.

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Classical Semiconductors:
Gallium Arsenide

Head Dr. Christiane Frank-Rotsch
Team Dr. N. Dropka, Dr. K. Giziewicz, O. Root, Dr. R. Zwierz

Überblick

Der III-V-Verbindungshalbleiter Galliumarsenid (GaAs) ist neben Silicium das am häufigsten eingesetzte Halbleitermaterial. GaAs wird häufig für die drahtlose Kommunikation, sowie in der Mikrowellen- und in der Hochfrequenztechnologie eingesetzt, dies führte zu einem starken Anstieg des GaAs-Markts in den letzten Jahren. Das starke Wachstum des GaAs-Marktes in den letzten Jahren ist vor allem auf die Entwicklung der Mobilfunk- und LED-Märkte zurückzuführen, einschließlich der 3D-Bildgebung auf Basis von VCSELs, die besonders stark zugenommen hat. Gegenwärtig steigt die Bedeutung von GaAs auch als Materialbasis für Detektoren. Im Fokus der Forschungsaktivitäten steht, neben der fortlaufenden Verbesserung der Qualität der Kristalle, eine Effizienzsteigerung beim Kristallisationsprozess sowie die Absenkung von Prozesskosten. Eine wesentliche Voraussetzung für die Züchtung von VGF-GaAs-Einkristallen hoher Qualität ist eine leicht konvexe, nahezu flache Form der fest-flüssig Kristallisationsfront. Der Einsatz von externen Feldern, insbesondere von Wandermagnetfeldern (TMF) zeigt ein großes Potenzial, diese Anforderung zu erfüllen.

Die am IKZ in enger Zusammenarbeit mit akademischen und industriellen Partnern entwickelten KRISTMAG®-*Technologie [1] bietet ein leistungsstarkes Werkzeug, um definierte Wandermagnetfelder in einem breiten Parameterbereich zu erzeugen. Die* KRISTMAG®-*Technologie wurde bereits erfolgreich für die Züchtung von GaAs eingesetzt und in den letzten Jahren kontinuierlich weiterentwickelt.*

Derzeit sind zwei Vertical Gradient Freeze (VGF) Anlagen mit einem KRISTMAG®-*Heizer-Magnet-Modul (HMM) ausgestattet, das eine gleichzeitige, aber unabhängig regelbare Erzeugung von Wärme- und Wandermagnetfeldern ermöglicht. Die Forschungsaktivitäten der GaAs-Gruppe fokussieren sich vor allem auf die weitere Verbesserung der strukturellen Perfektion der VGF-Einkristalle sowie auf die Reduzierung von Temperaturfluktuationen während des Kristallwachstumsprozesses zur Stabilisierung des Einkristallwachstums.*

Zusätzlich wurde ein Decken-Heizermagnetmodul (THMM) [2] entwickelt, welches oberhalb des Tiegels positioniert wird. Das THMM ermöglicht eine effektive Beeinflussung der Form der fest-flüssig-Phasengrenze besonders bei zunehmender Kristalllänge. Die meisten der Forschungsaktivitäten der Gruppe GaAs werden in enger Zusammenarbeit mit industriellen Partnern durchgeführt und unterliegen daher Geheimhaltungsauflagen.

Das DFG-Projekt „Modellbasierte Steuerung und Regelung des Vertical-Gradient-Freeze-Kristallisationsprozesses mit Hilfe verteilparametrischer Methoden“ fällt nicht in diese Kategorie und dessen Ergebnisse können im Detail vorgestellt werden. In diesem DFG-Projekt, welches in Kooperation mit dem Institut für Regelungstheorie der TU Dresden und dem Leibniz Institut für Katalyse bearbeitet wird, werden in einer numerischen Studie künstliche neuronale Netze (ANN) zur Optimierung der VGF-GaAs Prozessparametervorgaben (Rezept) angewendet.

Die Optimierung dieser Rezeptvorgaben zielt darauf ab dynamische Profile der Leistungen der Heizer zu ermitteln, die es ermöglichen, die gewünschte Temperaturverteilung in der Schmelze und im Kristall während des Prozesses zu erreichen und einzuhalten. Gängige Optimierungsmethoden, die bei der Kristallzüchtung verwendet werden, wie beispielsweise die inverse Modellierung, sind für dynamische Modelle und zahlreiche Variablen nicht anwendbar.

Overview

The III-V compound semiconductor gallium arsenide (GaAs) is the most widely deployed semiconductor material aside from silicon. GaAs is commonly used for wireless communication in the microwave or high-frequency technology. Strong growth of the GaAs market in recent years is caused mainly by the development of mobile telecommunications and LED markets, including 3D imaging based on VCSELs that has gained a particular significance. Recently GaAs has also become increasingly important as detector material.

Besides continuous improvements in crystal quality, the enhancement of the yield of the crystal growth process and the reduction of process costs are still our key research activities. One of the prerequisites for a high-quality Vertical Gradient Freeze (VGF) grown GaAs single crystal is a slightly convex, nearly flat shape of the solid-liquid (s/l) interface. The use of external fields, especially traveling magnetic fields (TMF), shows great potential to fulfill this requirement.

The KRISTMAG® [1] technology developed at IKZ in close cooperation with academic and industrial partners provides a simple yet powerful tool to create traveling magnetic fields within a broad parameter range. It has been successfully applied to the growth of GaAs and further developed in recent years.

Currently, two VGF furnaces are equipped with a KRIST*MAG*® heater magnet modules (HMM), allowing simultaneous but decoupled generation of heat and traveling magnetic fields. The research activities in the GaAs group are mainly focused on the further improvement of structural perfection of VGF single crystals, as well as on the reduction of temperature fluctuations during the process to stabilize the single crystal growth. Additionally, to effectively influence the shape of the s/l interface in longer crystals, a top HMM (THMM) [2] has been developed and brought into operation. Most research projects in the GaAs group are conducted in close cooperation with industry partners and are therefore subject to confidentiality.

The DFG project "Model-based control and regulation of the VGF crystal growth process using distributed parametric methods" in cooperation with the Institute of Control Theory at TU Dresden and the Leibniz Institute for Catalysis at the University of Rostock does not fall into this category, thus can be described in this report.

Within the framework of this DFG project, we performed a feasibility study on the use of artificial neural networks (ANNs) to optimize the VGF-GaAs growth recipe. Recipe optimization will help to find the dynamic profiles of power supply that enable reaching and keeping desired temperature distribution in the melt and crystal during the run. Common optimization methods used in crystal growth such as inverse modeling are not applicable to dynamic models and numerous variables.

Results

The application of TMF to enhance the VGF growth of single GaAs crystals has been one of the group's core activities in the last few years. The continuous improvement of KRIST*MAG*® technology is marked by the numerous achievements and has been partially covered in previous annual reports.

The flattening of the solid/liquid (s/l) interface through a precise and constant control of the melt flow during the growth has been one of the key results of our research. This enabled us to achieve a higher growth rate for VGF high-quality crystals [3].

In 2016, we started the joint project EneRed with an industrial partner, Freiberger Compound Materials GmbH. This project aims to reduce the energy required for single crystal growth e.g., by an increased growth rate. First 4 inch GaAs single crystals were grown using the newly developed top HMM (THMM). Two spiral heaters placed above the crucible enable generation of downward Lorentz forces oriented outwards (Fig. 1 (b)) that reduce concavity in the region near the crucible wall, typical for the W-shape s/l interface (Fig. 1 (a)). For details, please, see the IKZ Annual Report 2016.

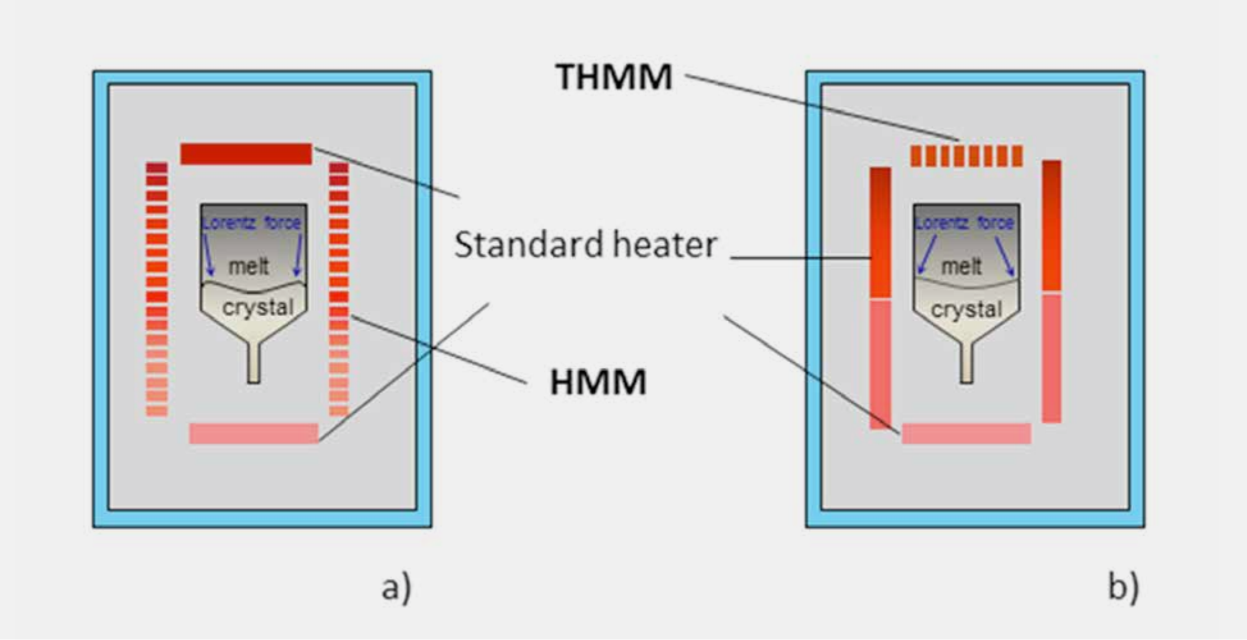

Fig. 1
Sketch of heater arrangement: a) with side HMM b) with THMM.

Optimized TMF parameters were confirmed by numerical simulations while setting suitable thermal conditions for the growth remains challenging due to the heater's proximity to the melt. Nevertheless, a big advantage of THMM is that its installation in a VGF furnace requires only minor changes in the hot zone, facilitating future transfer to industry. A further aim of the investigation of the growth process is to achieve uniform crystallization rate. During process development, a temporary alteration of the magnetic field is used to mark the s/l phase boundary. This method, already described in detail in the respective patent [2] and IKZ Annual Report 2012, does not interrupt the growth process.

Fig. 2
Image of 4 inch VGF GaAs single crystal crystallized in Top HMM with 9 markers (marked with red arrows).

Figure 2 shows an image of a 4 inch VGF GaAs crystal grown using THMM. 9 TMF-induced-markers indicate the growth interface. A nearly uniform distance between the TMF markers confirms a constant growth rate. We continue to investigate the growth process optimization using THMM, and in the next year will focus on a further increase of growth rate. Furthermore, the targeted use of magnetic fields offers an opportunity to influence the defect concentration of semiconductor crystals. This resulted in new project ideas related to defect engineering that have already been leveraged in cooperation with different international partners. In this context, we would like to highlight a recently commenced cooperation with the Lawrence Berkeley National Laboratory.

The DFG project "Model-based control and regulation of the VGF crystal growing process using distributed parametric methods" started in April 2016, has successfully advanced in 2017. In this study, we focused on a dynamic ANN as an effective method for forecasting time series events that can be used in the next step for optimization. Memory is the most attractive feature of a dynamic ANN. Its response at any given time depends not only on the current input, but on the history of the input sequence. Therefore, a dynamic ANN is suitable for quick point-wise prediction of unknown functions. Particularly, we correlated the dynamic profiles of the power of heaters (2 inputs x_i) with the temperature at various control points in a GaAs melt and the position of s/l interface (6 outputs y_i). In the course of this feasibility study, 500 growth recipes (temporal profiles of x_i and y_i) for the derivation of a dynamic ANN were generated by transient 1D simulation of the VGF-GaAs growth. The best ANN predictions of dynamic profiles of temperature in various positions in GaAs melt and crystal, as well as the position of s/l interface, were obtained using an ANN architecture with one hidden layer with 9 neurons, as shown in Figure 3.

The first results obtained on the application of monolayer dynamic ANN for forecasting the crystal growth cooling program were promising (Fig. 3), and the first publication [4] attracted broad interest. However, for the application in advanced process control in smart factories (Industry 4.0), very accurate predictions are needed. To reach this goal, we continued our study and focused on more complex multilayer dynamic ANNs. This work is in progress.

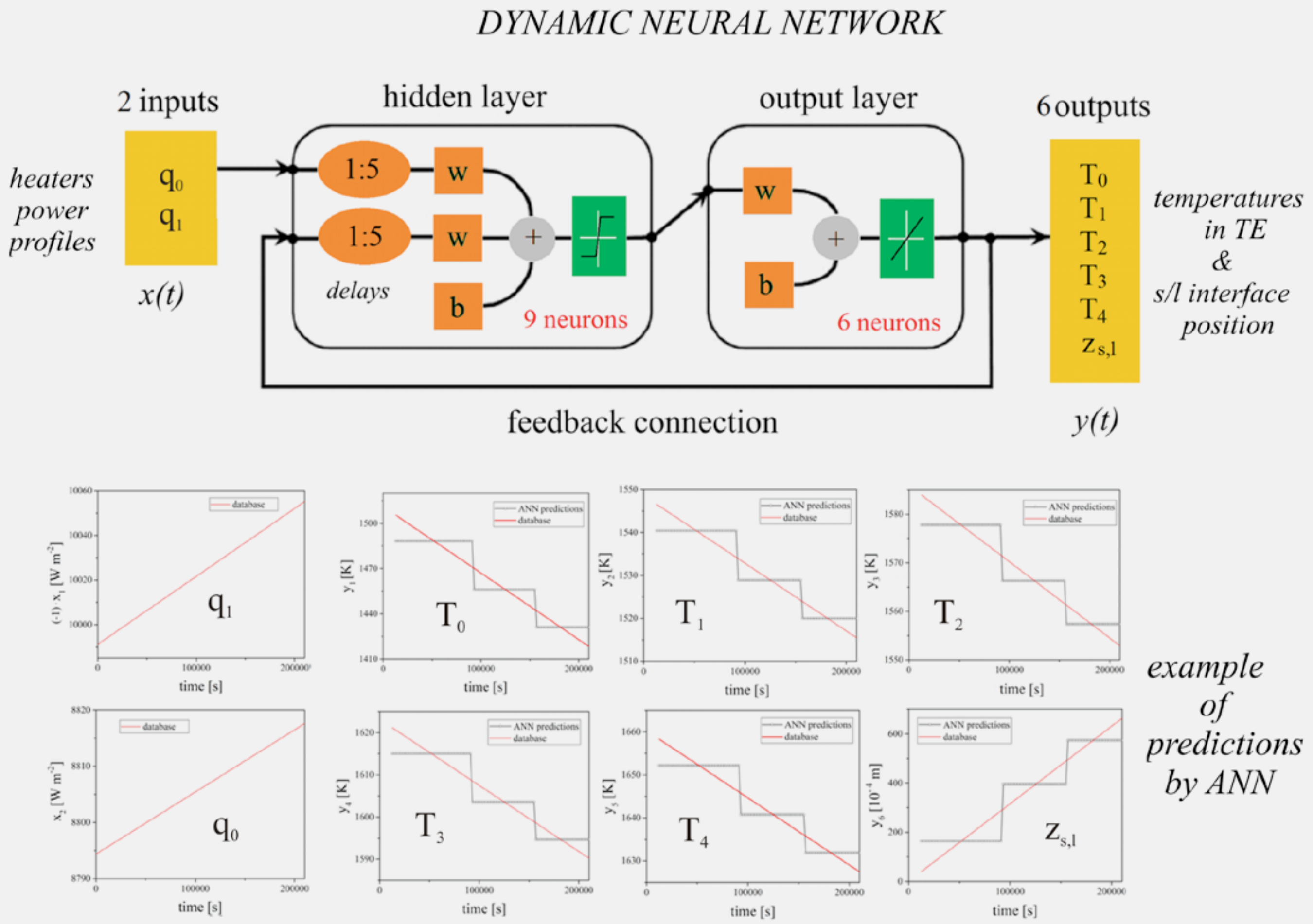

Fig. 3
Architecture of a dynamic ANN with 2 inputs, 6 outputs, and one hidden layer together with an example of predictions of dynamic temperature profiles and interface position by ANN. Arrows and lines in upper figure represent information flow.

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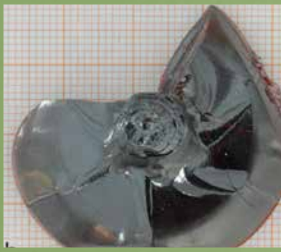
Dielektrika & Wide Bandgap

Dielectric & Wide Bandgap Materials



Dielektrika & Wide Bandgap

Head of department: Prof. Dr. Matthias Bickermann

Die Abteilung ist spezialisiert auf die Kristallzüchtung von Nitrid-, Oxid- und Fluorid-Massivkristallen.
Sie untersucht verschiedene Materialien und entwickelt Einkristalle mit maßgeschneiderten Eigenschaften und hoher Ausbeute, die als Substrate für neuartige energieeffiziente, elektronische und optoelektronische Bauelemente eingesetzt werden können. Darüber hinaus werden Oxid- und Fluoridkristalle für Laser- und nichtlineare optische Anwendungen bereitgestellt.

Ziel der Abteilung ist es, Einkristalle mit höchster struktureller Qualität zu züchten und materialspezifische Wachstumstechnologien für den industriellen Transfer zu entwickeln. Der Schwerpunkt liegt auf der Kontrolle der elektrischen und optischen Eigenschaften durch geeignete Techniken des Wachstums, der Dotierung und der Charakterisierung.

Durch Kooperationen und Dienstleistungen für Unternehmen und Forschungseinrichtungen stellt die Abteilung die Materialbasis für viele Forschungsprojekte innerhalb und außerhalb des IKZ zur Verfügung.

Aktuelle Arbeitsgruppen in der Abteilung sind:
- *Oxide & Fluoride*
- *Aluminiumnitrid*

Dielectric & Wide Bandgap Materials

The department specializes in bulk crystal growth of nitrides, oxides, and fluorides. It evaluates different materials and develops single crystals with tailored properties and high yield for use as substrates in novel energy-efficient electronic and optoelectronic devices. In addition, oxide and fluoride crystals are prepared for laser and non-linear optical applications.

The department targets to prepare bulk crystals with exceptional structural quality and to develop material-specific growth technologies ready for industrial transfer. The special focus is on the control of electrical and optical properties through suitable techniques of growth, doping, and characterization.

Through collaborations and service to companies and research institutions, the department provides the materials basis for many research projects inside and outside IKZ.

Current working groups in the department are:
- **Oxides & Fluorides**
- **Aluminium Nitride**

Dielectric & Wide Bandgap Materials:
Oxides & Fluorides

Head Dr. Steffen Ganschow
Team M. Brützam, Dr. Z. Gałązka, Dr. C. Guguschev, M. Rabe, A. Tauchert, Dr. D. Schulz, I. Schulze-Jonack, E. Thiede

Überblick

Die Arbeitsgruppe bearbeitet schwerpunktmäßig Forschungsaufgaben zur Schmelzzüchtung von Oxid- und Fluorideinkristallen. Arbeitspferd der Kristallzüchtung ist das weit verbreitete Czochralskiverfahren zur Herstellung großer Einkristalle mit hoher struktureller Perfektion und chemischer Reinheit. Unsere apparative Ausstattung ermöglicht die Züchtung von Kristallen mit Schmelzpunkten von bis zu 2200 °C in verschiedenen Atmosphären, von stark reduzierend bis oxidierend. Dabei sind Kristallgrößen von bis zu etwa 50 mm Durchmesser und 150 mm Länge erzielbar.

Die Czochralskimethode ist jedoch streng genommen nur für kongruent schmelzende Substanzen anwendbar. Viele relevante Materialien erfüllen diese Bedingung jedoch nicht oder haben einen kongruenten Schmelzpunkt, der nicht mit der ganzzahligen „idealen" Stöchiometrie zusammen fällt. In diesen Fällen muss zunächst das Kristallisationsverhalten untersucht und daraus geeignete Bedingungen für die Züchtung abgeleitet werden.
Das geschieht in enger Kooperation mit der Gruppe Chemische & Thermodynamische Analyse, die hierfür erforderliche Analysemethoden betreibt. In Abhängigkeit von den konkreten Anforderungen des Materialsystems kommen unter Umständen auch andere Schmelzzüchtungsmethoden wie die EFG-Methode (edge-defined film-fed growth), die Bridgman- und die Kyropoulos-Methode zum Einsatz. Inkongruent schmelzende Verbindungen werden meist aus der Schmelzlösung nach dem TSSG-Verfahren hergestellt, und bei hoher Flüchtigkeit kann auch die Züchtung aus der Gasphase die Methode der Wahl sein.

Im Berichtszeitraum wurde unser Anlagenpark um zwei neue Anlagen erweitert. Zwei von drei im Rahmen eines vom BMBF geförderten Projektes („EQuiLa" – Erforschung und Qualifizierung innovativer Lasermaterialien und -kristalle) zur Etablierung des Zentrums für Lasermaterialien am IKZ neu erworbenen Anlagen konnten in Betrieb genommen werden: eine Czochralski-Anlage, die den mit dem Zentrum entstandenen zusätzlichen Bedarf nach optischen Kristallen zu befriedigen hilft, sowie eine Vier-Spiegel-Zonenschmelzanlage mit Halogenlampen als Wärmequelle. Diese erweitert das Methodenspektrum der Arbeitsgruppe in einem entscheidenden Punkt, denn sie ermöglicht erstmals die Züchtung stabförmiger Einkristalle aus der Schmelze ohne Einsatz eines Tiegels. Damit einher geht die Möglichkeit, Materialien unter stark oxidierenden Bedingungen bis hin zum Einsatz reinen Sauerstoffs zu schmelzen.

Das Tätigkeitsfeld der Gruppe erfuhr auch eine thematische Erweiterung. Mit den Arbeiten in zwei BMBF-Projekten „EQuiLa" und „IsoNova" (Entwicklung von Faraday-Rotatoren mit stark verbesserten Eigenschaften auf der Grundlage von Kalium-Terbium-Fluorid (KTb_3F_{10}) und anderen innovativen Materialien) bilden optische Materialien seit Längerem wieder einen wichtigen Schwerpunkt. Im Rahmen von EQuiLa werden Seltenerd- und Übergangsmetall-dotierte oxidische und fluoridische Kristalle hergestellt und strukturell und spektroskopisch in Hinblick auf ihre Eignung für den Laserbetrieb charakterisiert. Die Konsortialpartner des IsoNova-Projektes (IKZ, FEE, Fraunhofer IPM, TRUMPF und TOPTICA) decken die gesamte Wertschöpfungskette von der Entwicklung der Kristallzüchtung bis zur Evaluierung kompletter Isolatoren im Laser-Dauertest ab. Am Anfang dieser Kette stehen die Arbeiten unserer Arbeitsgruppe, die Züchtung von KTb_3F_{10} Einkristallen, die den hohen Anforderungen der angestrebten Anwendung genügen.

Neben diesen neuen spannenden Aufgaben führten wir die Arbeiten zu etablierten Schwerpunktthemen fort:
- *Kristalle mit kubischer und pseudokubischer Perowskitstruktur für die Anwendung als Substrate für die epitaktische Abscheidung multifunktionaler Schichten,*
- *transparente halbleitende Oxide,*
- *neue piezoelektrische Oxidkristalle für die Anwendung in Druck- oder Kraftsensoren unter extremen Bedingungen und*
- *Untersuchungen zum Wachstum von Delafossit-Kristallen im Rahmen eines DFG-Projektes.*

Neben diesen Schwerpunktaufgaben wurden auch im Berichtszeitraum wieder zahlreichen Forschungsinstituten oxidische und fluoridische Einkristalle zur Verfügung gestellt, teilweise mit dem Ziel der zukünftigen Einreichung gemeinsamer Projektanträge.

Overview

The group works on scientific and technological issues associated with the melt growth of oxide and fluoride single crystals. The workhorse of crystal growth is the widely used Czochralski technique, which involves pulling of bulk single crystals of high structural perfection and chemical purity. Our equipment allows to melt materials at temperatures above 2200 °C in various ambient spanning from strongly reducing to slightly oxidizing. Crystals of more than 50 mm diameter and 150 mm length can be achieved.

Application of the Czochralski technique primarily requires congruent melting behavior of the substance to be grown. However, numerous crystals of scientific or technological importance do not fulfill this condition or have a congruent melting composition that does not coincide with the ideal integer stoichiometry. In these cases, we have to study the melting behavior and derive appropriate conditions for the growth of single crystals before the experiments. Here, we work very closely with the "Chemical and Thermodynamic Analysis" group operating essential analytical and computational methods. Depending on the constraints imposed by the material system, growth methods other than the Czochralski technique may be applied, e.g., the edge-defined film-fed growth (EFG) method, the Bridgman, and the Kyropoulos techniques. Incongruently melting compounds are mainly grown from a high-temperature solution, and, in case of high volatility, growth from the gas phase using an appropriate species transport may be the method of choice.

In the reporting period, we increased our capacity by two new growth stations: a modern Czochralski puller to meet the growing demands of the Centre for Laser Materials and a four-mirror optical-zone melting apparatus that extends the range of available growth methods to include a crucible-free one. Moreover, the equipment allows to melt materials in strongly oxidizing atmosphere, e.g., in pure oxygen. The systems were obtained through the financial support from the funds of the BMBF project "EQuiLa" (Erforschung und Qualifizierung innovativer Lasermaterialien und –kristalle; Research and qualification of innovative laser materials and crystals).

The group started two new activities with a focus on optical materials supported by BMBF projects. The „EQuiLa" project focuses on research and evaluation activities of innovative laser host materials based on rare earth and transition metal doped oxide and fluoride single crystals. The BMBF project „IsoNova" (Entwicklung von Faraday-Rotatoren mit stark verbesserten Eigenschaften auf der Grundlage von Kalium-Terbium-Fluorid (KTb_3F_{10}) und anderen innovativen Materialien; Development of Faraday rotators with greatly improved properties on the basis of potassium terbium fluoride (KTb_3F_{10}) and other innovative materials) aims to develop new efficient Faraday rotator materials for use in optical isolators. The consortium partners of the project cover the entire value chain from crystal growth up to the assessment of isolators in endurance tests. Being at the very beginning of the chain, crystal growth provides the base for all subsequent activities and is therewith essential for the further course of these projects.

Besides these new exciting challenges, we continued the work in well-established topics:

- crystals with (pseudo)cubic perovskite structure as substrates for the epitaxial growth of functional films;
- transparent semiconducting oxides;
- new piezoelectric crystals for transducers in sensors working at high temperature and harsh environments;
- investigation on the growth of delafossite crystals in the frame of a DFG-funded project.

In addition to these projects, in 2017 we provided oxide and fluoride single crystals and single crystal samples to numerous research institutes in the frame of scientific service.

Results

Large-lattice-parameter perovskite substrate crystals

Since the foundation of the Leibniz Institute for Crystal Growth perovskites have been continuously researched and developed. Single crystals in the form of epi-ready substrates have served as the basis for the development of advanced epitaxially grown films with exciting ferroelectric, superconducting, ferromagnetic, piezoelectric, multiferroic, or electronic properties. Due to increased interest of scientific communities from all over the world and of the multi-institutional Leibniz ScienceCampus GraFOx, we currently focus on the crystal growth development of novel perovskites with large cubic or pseudo-cubic lattice parameters above 4 Å.

In particular, lattice-matched substrates are of high relevance to enable heteroepitaxial growth of innovative high-quality thin films like $(Ba,La)SnO_3$, $BiScO_3$ or $PbZrO_3$ with interesting semiconducting, piezoelectric and antiferromagnetic properties, respectively. During our previous studies, we identified the solid solution system $(LaLuO_3)_{1-x}(LaScO_3)_x$ as suitable substrate material for these layers [1]. With the growth of bulk crystals, perovskite substrates for the pseudo-cubic lattice parameter range 4.12 – 4.15 Å were provided for the first time. To enable the epitaxy of $(Ba,La)SnO_3$ on substrates with slightly lower lattice parameters, a novel substrate crystal was grown by expanding the ternary system La_2O_3 – Sc_2O_3 – Lu_2O_3 to a quaternary system, i.e. a further rare earth oxide was added. We applied this approach and found, that a pseudo-cubic lattice parameter of about 4.086 Å is accessible (Fig. 1) by growing crystals at temperatures below the maximum thermal load of iridium crucibles (≈ 2250°C). Currently, novel double perovskites with nearly perfect lattice match are under investigation.

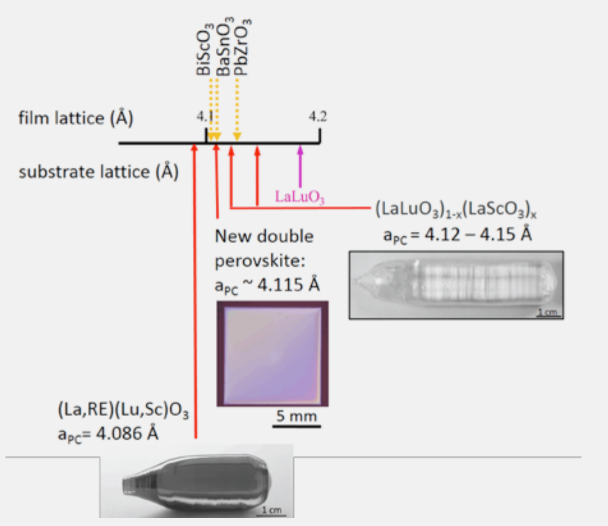


Fig. 1

Selection of substrate crystals and thin films with pseudo-cubic or cubic lattice parameters above 4 Å.

Czochralski growth of high-quality $Tb_2Ti_2O_7$ single crystals with pyrochlore structure

Melt-grown bulk terbium titanate ($Tb_2Ti_2O_7$) single crystals were recently a subject of a feasibility study carried out in cooperation with the Forschungsinstitut für mineralische und metallische Werkstoffe -Edelsteine/Edelmetalle GmbH (FEE) and the Cornell University. Terbium titanate is a spin-ice material with remarkable magneto-optical properties. It has a high Verdet constant and is a promising substrate crystal for the epitaxy of quantum materials with pyrochlore structure. So far, large single crystals of $Tb_2Ti_2O_7$ or any oxidic pyrochlore with adequate quality are not commercially available. Here we report on the growth of high-quality bulk crystals using the Czochralski method.

Previous work using the automated Czochralski method has suffered from growth instabilities like diameter fluctuation, foot formation, and subsequent spiraling shortly after the seeding stage. We substantially suppressed growth instabilities resulting from the limited heat flow through the melt/crystal interface by a combination of manual growth control, moderate to high pulling rates, and sufficient undercooling of the melt [2]. Thereby, the volumes of the crystals were strongly increased to several cubic centimeters, resulting in crystal diameters between 30 mm to 40 mm and crystal lengths of up to 10 mm (Fig. 2). We evaluated the crystalline quality by using rocking curve X-ray diffraction (XRD) measurements on as-grown {111} facets with a high-resolution diffractometer. The rocking curve measurements revealed full width at half maximum values between 28 and 40" for 222 reflections. Such high crystal quality is demonstrated for the first time, and the experience gained from these experiments constitutes a good starting point for future crystal growth developments in the field of novel pyrochlores.

Preparation of KTb_3F_{10} crystals

An optical isolator is an essential component of many high power lasers. It allows light to travel in only one direction and attenuates light travelling in the backward direction thus protecting the laser from back-reflected radiation. The heart of an optical isolator is a Faraday rotator crystal, a crystal able to turn the plane of polarization of throughpassing light when placed in a strong magnetic field. The recent trend towards miniturization of lasers requires the development of compact optical isolators and by that the development of new highly efficient Faraday crystals. Prospective candidates of such are subject of a BMBF-funded project „IsoNova“.

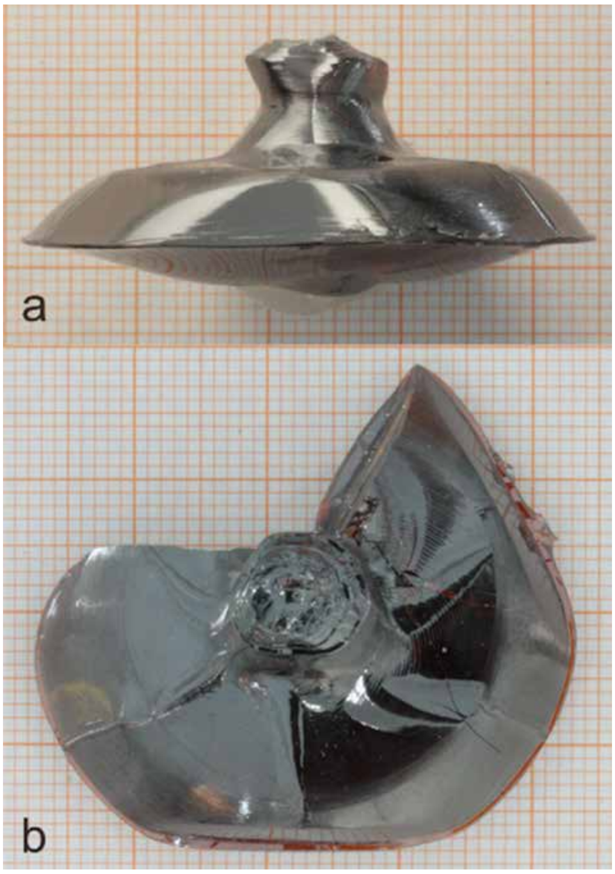


Fig. 2

As-grown $Tb_2Ti_2O_7$ Cz single crystal (length 10 mm, diameter 40 mm) shown from the side (a) and from the top (b), respectively. The crystal was grown at a rate of about 8 mm/h. The figure is reproduced from Ref. [2] with permission from the Royal Society of Chemistry.

At IKZ we investigate crystal growth of potassium terbium fluoride (KTb_3F_{10}, KTF) – a rediscovered fluoride with cubic structure and high Verdet constant (rotation angle per unit throughpassed length and magnetic field flux) and excellent transparency that promises outstanding isolator performance in the visible spectral range.

For the growth of optical grade fluoride single crystals it is essential to control not only the metal stoichiometry, e.g. the K:Tb ratio, but also the contamination of the raw materials by oxides and/or hyroxides and water. Upon heating already traces of moisture may cause hydrolysis and finally lead to the formation of insoluble oxides which affect the stability of the growth and may seriously diminish crystal quality.

To remove undesired non-metallic impurities, the raw materials usually have to undergo a thermal pretreatment in an reactive ambient, a step called fluorination. Fig. 3 shows DTA (differential thermal analysis) measurements that were performed in a NETZSCH STA 449C „Jupiter“. The upper two curves result from the first (solid line) and second (dashed line) heating of fresh „as delivered“ TbF_3. The melting peak that appears near 1172 °C is not shown in this figure. The small peak with onset near 960 °C is attributed by some authors to a transition between two solid phases of TbF_3, but this assumption is wrong. If at all such phase transition takes place, it happens significantly below 900 °C. This is indicated by the dotted line in the insert of Fig. 3; this sketch of phase equilibria in the TbF_3 rich part of the TbF_3—Tb_2O_3 system is based on an article by Sobolev et al. [4]. This article shows that also minor oxygen traces in the order of a few mol% result in the eutectoid formation of a hexagonal phase $TbF_{3-2x}O_x$. This phase formation at 966 °C can easily mixed up with a phase transition of TbF_3, which according to the same authors possibly does not exist et al. [5]. It is remarkable that the eutectoid impurity peak that is small in the first heating becomes significantly larger during the second heating: obviously oxygen/water traces in the argon flow with 99.999% purity that was used to rinse the DTA apparatus were sufficient to enhance the amount of oxygen impurities. Fortunately by treating the TbF_3 starting material with flowing HF gas we could obtain a significantly enhanced chemical quality. During the first heating of such hydrofluorinated TbF_3 no indication of the eutectoid formation of oxyfluoride were visible.

It should be noted, however, that superior purity of the environment must be maintained during the whole growth process, because already the second DTA heating run (without intermediate opening of the apparatus) showed again a minor trace of the peak near 960 °C.

At the moment there is no reliable phase diagram of the KF—TbF_3 system available and reports of different groups concerning the melting behavior of KTF are inconsistent. Neverthless, it appears that crystals must be grown from a melt with KF excess. We could perform few growth experiments that yielded several KTF boules. The obtained crystals are mainly opaque and possibly polycrystalline in major parts. However, transparency is slightly enhanced when pretreated material is used and individual grains across the diameter of a crystal boule have similar crystallographic orientation approximately 15 degree off [110]. To achieve optical quality of the grown crystals we will in the next step clarify the nature of scatterers that lead to apparent opacity and based upon these findings further optimize the fluorination process.

Transparent semiconducting oxides (TSOs)

Bulk TSOs single crystals constitute a crucial part of a diversity of IKZ’s running projects and collaborations, including ScienceCampus GraFOx, funded by the Leibniz Association, as well as other projects funded by the DFG, in the frame of the Leibniz Competition, and BMBF. The activity includes the growth research and development, as well as an extensive internal and external characterization (optical, electrical, thermal, mechanical, and structural properties) of various bulk TSO single crystals: β-Ga_2O_3, In_2O_3, SnO_2, $MgGa_2O_4$, $CoGa_2O_4$, $ZnGa_2O_4$, and $BaSnO_3$, as shown in Fig. 4. An extended portfolio of unique bulk TSO crystals (most of them available only at IKZ) offers a wide spectrum of crystal structures, optical, and electrical properties that may boost development of novel electronic and optoelectronic devices.

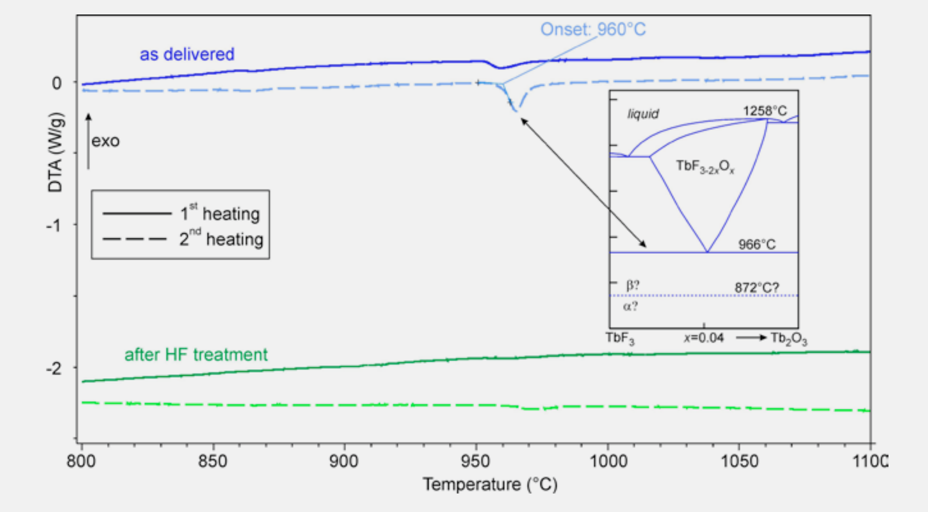


Fig. 3
DTA measurements of different charges of TbF_3 (delivered by Fox chemicals Ltd.).
The inset shows the TbF_3 edge of the TbF_3—Tb_2O_3 system.

Among bulk single crystals of the TSOs being under research and development at IKZ, β-Ga_2O_3 is the most highly demanded crystal material for both fundamental research and substrates for homepitaxy, in particular, by MOVPE in-house (see report or group Semiconducting Oxide Layers). Such structures define a platform for high power devices and UV optoelectronics. Bulk crystals of β-Ga_2O_3 are grown by the Czochralski method at two diameters: 2 cm mainly for doping purposes, and 2 inches for substrates. Doping and co-doping include Mg, Si, Sn, Cr, Al, Ce, Ce, and Ce+Si [6] for tuning optical/electrical properties, and for studying scintillation properties under joint project GoScint DFG (IKZ) – NCN (Nicolaus Copernicus University, PL) that extends the capability of detection of nuclear radiation by Ga_2O_3, in addition to demonstrated detection of fast neutrons [7]. The growth of 2 inch diameter crystals with further scale-up capabilities combined with unique electrical / optical proeprties constitite prerequistes for industrial scale production of bulk β-Ga_2O_3 crystals, layers, and devices. The electrically insulating substrates at room temperature are achieved by Mg-doping, very high oxygen concentration during growth and cooling down, or a combination of both. High or very high oxygen concentration during growth is the sine qua non condition for growing large volume β-Ga_2O_3 crystals, which is realized by our novel approach of oxygen supply to a growth furnace involving an Ir crucible [8, 9]. That new approach brings the growth technology of β-Ga_2O_3 crystals to a new level in terms of structural perfection and further scale-up capabilities. Moreover, an exploration of thermal and mechanical properties of β-Ga_2O_3 enabled to develop a numerical model for stress generation in a growing crystal [10] that becomes important when the scale-up is in quest.

The β-Ga_2O_3 substrates are used in several projects, including DFG, VIP+, and AFL, which aim is to study fundamental properties, epitaxy, and construction of devices for high power electronic applications [11, 12]. The substrate preparation and orientation was found to be critical for the structural quality and electrical properties of epitaxial layers [13], and therefore requires a special attention. The substrates are also used to grow homoepitaxial layers by molecular beam epitaxy (MBE) at Paul-Drude-Institut für Festkörperelektronik (PDI) [14] and by liquid phase epitaxy (LPE) at Kazimierz Wielki University (UKW) in Poland, to form nanostructures at Helmholtz-Zentrum Berlin (HZB), and for LEDs fabrication at Ferdinand-Braun-Institut (FBH) [15]. We also collaborate with Surrey University (UK) and Nebraska-Lincoln University (US) for fundamental studies of potential applications of β-Ga_2O_3 in the THz wavelength range and its dielectric function, respectively.

Within ScienceCampus GraFox activity, fundamental surface, optical and electrical properties of In_2O_3 and SnO_2 are continuously investigated [16, 17]. Besides, for the first time In_2O_3 will be used as substrates for homoepitaxy at PDI and for application in gas sensors.

Another important group of materials is Ga-based spinels, $MeGa_2O_4$, where Me is a divalent metal ion. Earlier we demonstrated that $MgGa_2O_4$ is an ultra-wide bandgap oxide semiconductor, however, the carrier mobility is quite low. $MgGa_2O_4$ is under detailed study in collaboration with Humboldt-Universität Berlin, Ruhr-Universität Bochum, and Technische Universität Berlin. Now we introduce truly bulk single crystals of a new Ga-based spinel, $ZnGa_2O_4$, which we grew from the melt for the first time. Its electrical and optical properties are similar to β-Ga_2O_3, but the mechanical properties are better due to cubic structure (no easy cleavage planes).

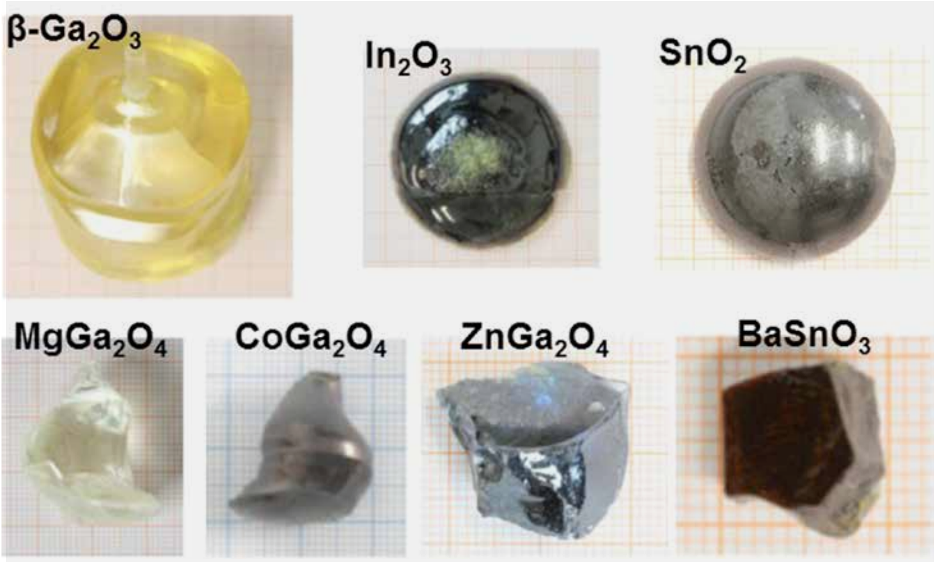


Fig. 4
Bulk single crystals of various TSOs.

Despite of high thermal instability of the material, high melting point (1900 °C), and incongruent evaporation, we have obtained single crystals of 3 cm^3 at an early stage of the growth study with capabilities of further scaling-up. $ZnGa_2O_4$ will be used for homoepitaxial growth and construction of electronic power devices in collaboration with Seoul National University. In addition to semiconducting properties, Ga-based spinels have good lattice match with ferrite spinels. It has been demonstrated, in collaboration with Alabama University, that the use of our $MgGa_2O_4$ and $CoGa_2O_4$ substrates eliminate anti-phase boundaries in nickel ferrite ($NiFe_2O_4$) thin films [18]. $ZnGa_2O_4$ has almost ideal lattice match with $NiFe_2O_4$. Availability of Ga-based spinels resulted in high interest in this class of materials and extensive collaboration, including Air Force Research Laboratory and Stanford University.

Our demonstration of obtaining bulk $BaSnO_3$ crystal samples from the melt for the first time [19] resulted in a project granted in a frame of Leibniz Competition ("BaStet") involving crystal growth, characterization, homoepitaxy, and heteroepitaxy, in collaboration with Paul-Drude-Institut für Festkörperelektronik (PDI), Technische Universität Berlin, and Humboldt-Universität Berlin. $BaSnO_3$ has the highest electron mobility among other TSOs which in combination with its perovskite substrate offers novel opportunities that are not available for other perovskites.

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Dielectric & Wide Bandgap Materials: Aluminium Nitride

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Überblick

Einkristallines AlN kann als natives Substratmaterial für Al-reiche AlGaN-Schichten verwendet werden, die man für die Entwicklung von effizienten UVC-Lichtemittern, Laserdioden und neuartigen elektronischen Leistungsbauelementen sowie Sensoren benötigt. Eine Voraussetzung sowohl für die Technologieentwicklung als auch für die industrielle Nutzung ist die Bereitstellung von AlN-Wafern mit reproduzierbaren und definierten Eigenschaften sowie verwertbarer Größe. Vor diesem Hintergrund hat die Gruppe AlN des IKZ im Rahmen des vom BMBF geförderten Konsortiums „Advanced UV for Life“ die Arbeiten zur Herstellung von AlN-Volumenkristallen durch physikalischen Gasphasentransport (physical vapor transport - PVT) weitergeführt und das gemeinsame Forschungsprojekt „AlN-Substrate“ erfolgreich abgeschlossen. Für 2018 ist ein weiteres gemeinsames Forschungsprojekt mit der Technischen Universität Berlin, dem Ferdinand-Braun-Institut und den Firmen FCM und CrysTec geplant. Ziel dieses Projekts ist es, die Technologie für das AlN-Kristallwachstum und die Substratpräparation weiterzuentwickeln und die Anwendung von AlN-Substraten für Wellenlängen im tiefen UV-Bereich um 230 nm zu erforschen.

Im Fokus stand die Verbesserung der Reproduzierbarkeit, der Ausbeute und der zuverlässigen Kontrolle der AlN-Eigenschaften während des Kristallwachstums. Dabei wurden AlN-Volumenkristalle in zwei Spezifikationen realisiert. Die erste zeichnet sich durch eine hohe Transparenz im tiefen UV-Bereich bei einer Wellenlänge von 265 nm aus (Absorptionskoeffizient etwa 14 cm^{1}), während die zweite Spezifikation semiisolierende Eigenschaften bei Temperaturen von bis zu 1400 °C aufweist. Beide Arten von Kristallen werden auf Basis des Standardverfahrens hergestellt und weisen eine strukturelle Qualität entsprechend dem Stand der Technik (Versetzungsdichte < 10^4 cm^{-2}) auf. Die Ergebnisse erlauben es, das Anwendungsgebiet von AlN-Volumenkristallen auf Bauelemente für die Hochleistungselektronik und für piezoelektrische Sensoren zu erweitern, die bei Arbeitstemperaturen über 1000 °C einsetzbar sind. Für diesen Temperaturbereich grundlegende optische und Transporteigenschaften von AlN wurden im Rahmen eines DFG-Projekts in Zusammenarbeit mit Prof. Holger Fritze, TU Clausthal untersucht.

Ein weiterer notwendiger Schwerpunkt der AlN-Forschung ist die Entwicklung von Ansätzen zur Vergrößerung des Durchmessers der Einkristalle, die durch die Facettierung der Zylinderflächen der Kristalle und der Abnahme der Wachstumsrate mit der Wachstumszeit nur gering ausgeprägt ist. Darüber hinaus führen Spannungsfelder an den Kanten der wachsenden Kristalle und ungeeignete Wachstumsbedingungen zur Polygonisation von Versetzungen und anderen Strukturdefekten, die sich in das Kristallvolumen ausbreiten und die strukturelle Qualität verschlechtern. Um den Mechanismus der strukturellen Verschlechterung zu verstehen und Strategien zu dessen Vermeidung zu entwickeln, wurden verschiedene Modifikationen des Wachstumsprozesses getestet und eine umfangreiche Analyse der Defekte durchgeführt. Ungeachtet der erzielten Fortschritte, ist die weitere Bearbeitung der formulierten Arbeitspunkte notwendig, um durch stetige Vergrößerung der Kristalldurchmesser industriell relevante Werte von 1 bis 2 Zoll zu erreichen.

UV-Licht emittierende Dioden für den 300 nm-Bereich leiden immer noch an vergleichsweise geringen externen Quanteneffizienzen (EQE) von etwa 2-10 % und Emissionsleistungen im unteren mW-Bereich [1]. Die Untersuchungen zu Möglichkeiten der Abstimmung der Gitterparameter von AlN aus dem letzten Berichtszeitraum wurden daher fortgesetzt, um AlGaN-basierenden Bauelementen besser entsprechen zu können. Anlass sind hohe Dichten von Threading-Versetzungen in den aktiven Schichten (TDD > 10^9 cm^{-2}), die aus dem Fehlen von gitterangepassten Substraten resultieren. Um die Zahl der Threading-Versetzungen auf Werte unter 10^6 cm^{-2} zu verringern, was für industrielle LEDs notwendig ist, müssen die Schichten auf dem Substrat mit der erforderlichen Gitterkonstante pseudomorph verspannt (d. h. gitterangepasst) wachsen. Der Mischkristallbildung zwischen AlN und ScN bieten dafür einen geeigneten Lösungsansatz. Im aktuellen Berichtszeitraum wurde das Verständnis grundlegender chemo-physikalischer Prozesse und spezifischer Anforderungen an das ScAlN-Wachstum weiterentwickelt und ein reproduzierbares Wachstum von ScAlN-Einkristallen mit bis zu 0,8 at% Scandium in AlN demonstriert. Die strukturelle Qualität dieser Mischkristalle blieb dabei auf dem Niveau von reinen AlN-Einkristallen.

Ein BMWi-gefördertes ZIM-Verbundprojekt zur mikrowellen-plasmaunterstützten Abscheidung von AlN wurde in 2017 erfolgreich abgeschlossen. In diesem Projekt wurde gemeinsam mit Industriepartnern eine innovative Technologie zur Abscheidung grobkörniger AlN-Dickschichten mit hoher Reinheit entwickelt, die auf dem reaktiven Plasmaspritzen von AlN basiert. Details zu dieser Technologie wurden im letzten Jahresbericht vorgestellt.

Overview

Single crystalline bulk AlN can be used as native substrates for Al-rich AlGaN layers which are needed for the development of efficient deep UV light emitters, laser diodes, and novel power electronic devices as well as sensors. A prerequisite for both technology development and industrial use is to provide AlN wafers with reproducible and defined properties and applicable size. In this sense, the group AlN of the IKZ has continued the work on the preparation of AlN volume crystals by physical vapor transport (PVT) within the "Advanced UV for Life" consortium supported by the BMBF and successfully completed the joint research project "AlN-Substrate". A further joint research project together with the Technische Universität Berlin, Ferdinand-Braun-Institut and the companies FCM and CrysTec is planned for 2018. This project aims to refine the technology for AlN crystal growth and substrate preparation and to explore the application of AlN substrates for deep-UV wavelengths around 230 nm.

One important work objective was to improve reproducibility, yield, and reliable control of AlN properties during crystal growth. We obtained two types of bulk AlN crystals: the first one has high deep-UV transparency at the wavelength of 265 nm (absorption coefficient about 14 cm^{1}), while the second has semi-insulating properties at temperatures as high as 1400 °C. Both types of crystals are prepared in the frame of the standard process and have state-of-the-art structural quality (dislocation density $< 10^4$ cm^{-2}). These results allow expanding the field of application of AlN bulk crystals to components for high-performance power electronics and piezoelectric sensors, for which working temperatures beyond 1000 °C are essential. Within a DFG project, we examined the fundamental high-temperature optical and transport properties of this material in collaboration with Prof. Holger Fritze and his team from TU Clausthal.

Another target of AlN research is to develop approaches to increase the diameter of the single crystals. Crystal enlargement is tedious because the sidewalls become faceted and the lateral growth rate decreases with growth time. Moreover, strain fields at the edges of the growing crystals and inappropriate growth conditions lead to polygonization and formation of defects, which propagate into the crystal volume and worsen the structural quality. We have attempted various modifications of the growth process and performed a thorough analysis of defects to understand the mechanism behind structural deterioration and develop strategies to mitigate it. Although some progress was made, we continue working on this challenge to reach industrially relevant values of 1-2 inches by crystal diameter enlargement.

The team continued the research on tuning the lattice parameters of AlN to match AlGaN-based devices better. UV light-emitting diodes for the 300 nm range still suffer from comparatively small external quantum efficiencies (EQE) of around 2–10 % and emission powers in the lower mW range [1], due to high densities of threading dislocations in the active layers (TDD $> 10^9$ cm^{-2}) resulting from the lack of lattice-matched substrates. To decrease threading dislocations below 10^6 cm^{-2}, which is necessary for industrial LEDs, the layers must grow pseudomorphically strained (i.e., lattice-matched) on the substrate with the required lattice constant. We are focusing on bulk mixed crystal formation between AlN and ScN. This year, we continued to develop the understanding of fundamental chemo-physical processes and specific equipment requirements of ScAlN growth and succeeded in the reproducible growth of ScAlN with up to 0.8 at% scandium in AlN while preserving the structural quality of the single crystal.

Finally, we successfully finished a BMWi-supported ZIM collaborative project for microwave plasma-assisted deposition of AlN. In this project, we have developed an innovative technology based on reactive plasma spraying together with industrial partners. As a result, we obtained coarse-grained AlN thick layers with high purity. Details of this technology and our project were provided in the previous annual report.

Results

AlN – a continuous development

The AlN team has continued the preparation of large AlN bulk single crystals by physical vapor transport (PVT) using the N-polar growth on the (000-1) AlN facet in a graphite-containing setup. In the last years, we have developed this technology from an initial AlN wafer source by spontaneous nucleation of seed crystals in a tantalum carbide container (TaC) [2] to seeded growth of AlN single crystals which, cut into wafers, are subsequently used as seeds for the next generation of crystals [3].

The main work was performed in the frame of the joint research project "AlN-Substrate" whose mission was to evaluate the industrial feasibility of AlN growth by PVT. One of the investigated topics concerns the technical development. We designed a new type of AlN growth station as industrial prototype. The new type was constructed to provide reproducibility, usability, and maintenance in an industrial environment: modules can be assembled and mounted separately, seals are prepared for leak testing, and electromagnetic compatibility guidelines are respected.

We chose hot-zone materials by their longevity and robustness and made their geometry as simple and confined as possible because they have to be replaced after a fixed number of hours at growth conditions or depending on the accumulated wear. The growth stations are built at the IKZ workshop, only the SPS control is added by a company (Fig. 1). In 2017, we installed the second station and reached the goal that both stations provide similar and interchangeable results.

Fig. 1
Image of an AlN growth station built at the IKZ workshop.

A second topic was the delivery of AlN with defined properties. In the “Advanced UV for Life” framework focused on deep-UV LEDs, the transmission of the substrate at emission wavelength is of utmost concern, as the p-type contact on the opposite side is opaque and the light must be coupled out through the substrate. Unlike sapphire, AlN features significant absorption below band-gap in the wavelength region of interest due to the defects. Especially around 265 nm, the wavelength providing means for efficient disinfection, commercially available AlN substrates have a huge absorption associated with carbon. We have shown in detail that this absorption can be quenched by a proper relation and amount of oxygen and carbon atoms incorporated in the AlN bulk crystal. These impurities are controlled by the starting material’s purity, the process conditions (e.g., temperature, growth direction), and the presence of getter materials such as tungsten and tantalum carbide. Details have been provided in the previous year’s annual report as an IKZ highlight. Based on the understanding of defect formation and impurity compensation regimes, we have improved our process and now can reliably provide AlN samples with an absorption coefficient < 25 cm^{-1} at 265 nm in over 90% of the useable area. We also deliver an absorption mapping with every sample to our partners, which work on epitaxy and test structures on the substrates, to estimate the quantum efficiency of their test devices. In this context, it is particularly important that they can measure this light yield through the full wafer, without tedious flip-chip mounting and mechanical substrate back-thinning, which is required when processing commercial wafers. By changing the getter materials and adjusting the growth temperature, high-temperature semi-insulating AlN boules can be grown instead.

The optical properties of the AlN crystals are not the sole prerequisite for their potential substrate use. The structural quality of AlN substrates is an essential parameter for the epitaxy of deep-UV devices, as high dislocation densities (> 10^{7} cm^{-2}) in the active layers strongly decrease the quantum efficiency of LEDs due to increased non-radiative recombination [1]. For deep-UV devices with high Al content in the active AlGaN layers, dislocations also act as efficient carrier traps that hinder charge carrier transport to the quantum wells. Also, epitaxial step-flow growth is possible only at a certain off-orientation of the substrate [4]. To fulfill this condition, we have to avoid any small angle grain boundaries or strong lattice bending in crystals and substrates, which is also a requirement for proper wafering. The requirements for crystalline perfection of the epitaxial layers can be characterized by having X-ray rocking half-widths (XRC FWHM) in the range of 13-25 arcsec without secondary peaks. Only bulk AlN substrates provide this high crystalline perfection since AlN templates grown on foreign substrates are deteriorated due to lattice mismatch, different thermal expansion coefficient, and issues with chemical compatibility.

A crucial step to prepare structurally high-quality AlN crystals is to optimize seed fixation. Since AlN-based adhesives could not be considered because of their poor purity, a mechanical solution had to be developed. At the same time, sublimation of the seed edges, as well as possible deformation of the holder during the heating, make it difficult to predict the influence of geometry precisely. To better understand the processes at the seed holder, we performed numerical simulations (Virtual Reactor for AlN 7.4, STR GmbH) of the species transport and concentration fields at the seed.

Fig. 2

AlN substrates cut from a crystal with 12 mm diameter; left is the crystal part grown in the latest stage of the process; the back dot on the surface is merely an indicator of the Al-polar surface.

To optimize the thermal field at the seed, we employed a combination of radiation shields made from tungsten (strong direction-dependent heat transfer), massive elements (poor isotropic heat conduction at high temperatures) and cavities (very good heat conduction at high temperatures through radiative transfer) in the seed area. Concentrically arranged outlets lead to a reduction of the majority components and suppress parasitic nucleation next to the seed. The mechanical design of the seed holder parts has also been optimized. The installation is now self-centering and self-adjusting to avoid errors, and the reproducibility is significantly increased. A slightly convex temperature field is desired as this favors diameter expansion and growth without additional defects such as small-angle grain boundaries.

By using a seed holder with a 10 mm aperture diameter, crystals with diameters of about 12 mm have been grown reproducibly within one run. The crystals have an N-polar (0001) facet as growth surface and prismatic hexagonal {1010} facets. The expansion of the crystal occurs via the growth on these lateral facets. These crystals formed the basis for the supply of AlN substrate material during the "AlN substrate" project. Using exact off-cut of 0.1 ± 0.05° with respect to the Al-polar (0001) surface, our partners were able to provide AlN homoepitaxial layers by metalorganic vapor phase epitaxy (MOVPE) with a perfect monolayer-height step-flow pattern on the whole wafer surface (Fig. 4).

Still, diameter expansion during AlN growth remains problematic. The increase per growth run remains limited and the growth conditions to mitigate strain and parasitic growth at the crystal edges are not entirely reproducible due to the difficult evaluation of set-up material degradation. As a consequence, the problem of defect formation in subsequent generations has not been completely solved, limiting the currently achievable single-crystalline diameter to 17.5 mm. Overcoming this limitation is a primary working task of the planned second "Advanced UV for Life" project.

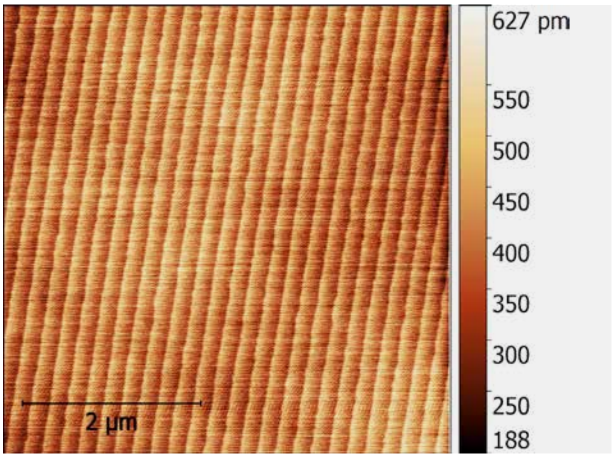

Fig. 4

AFM image of 5 µm thick AlN epitaxial layer on AlN bulk substrate, off-orientation 0.06° (image by C. Kuhn, TU Berlin, Institute of Solid State Physics).

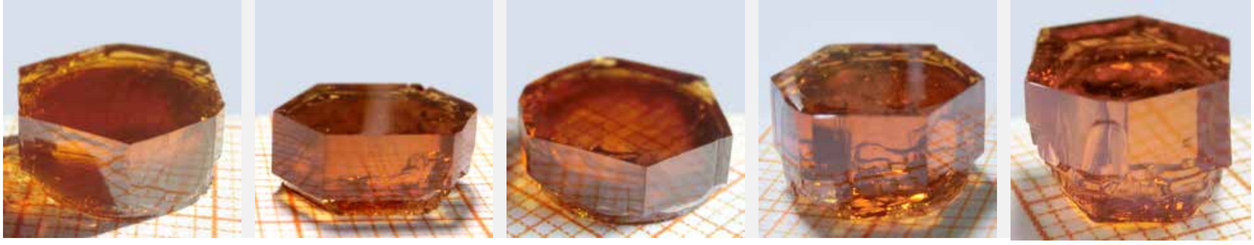

Fig. 3

Different AlN single crystals with diameters of 10-12 mm.

Growth of bulk $Al_{1-x}Sc_xN$ Single Crystals

A few years ago, Michelle Moram and coworkers predicted, by theoretical calculations, that $Al_{1x}Sc_xN$ substrates with $x \approx 0.08$ might exhibit lattice parameters that match those of $Al_{0.5}Ga_{0.5}N$, the compound used in devices emitting at about 300 nm [5, 6]. The minimum required scandium content might be even significantly lower, as a certain amount of residual compressive stress is allowed before relaxation leads to the generation of undesirable dislocations. E.g., the critical thickness for plastic relaxation of pseudomorphically strained layers already exceeds 1 µm for $Al_{0.7}Ga_{0.3}N$ on AlN substrates. So, while ScAlN thin films are researched with the focus on piezoelectric properties and GaN lattice matching, our focus is to provide bulk substrate materials of highest structural quality, however with a slight lattice constant increase with respect to AlN.

As PVT is the best method to grow single crystalline bulk AlN, it is also used to investigate a feasible growth technology for bulk ScAlN. To explore its strengths and limitations, the authors conducted growth experiments using different portions of ScN added to the AlN source material and (000-1) AlN wafers as seeds to obtain $Al_{1-x}Sc_xN$ single crystals at different seed temperatures in 600 mbar N_2. The aim was to combine AlN and ScN sublimation in order to prepare wurtzite $Al_{1-x}Sc_xN$ single crystals [7]. However, thermochemical calculations (FactSage [8, 9]) of the temperature-dependent ratio $p_{Sc}(g)/p_{Al}(g)$ shows only a tiny increase from 0.063 at 1950 °C to 0.079 at 2250 °C. While it should be possible to get $Al_{1-x}Sc_xN$ crystals with up to 8 at% by condensation of the vapor species, this means that the growth temperature can hardly influence the composition of the $Al_{1-x}Sc_xN$ crystals. In practice, our experiments have shown that factors such as segregation, solubility limits, and crystallographic effects (orientation dependent incorporation) as well as kinetic inhibitions play a decisive, and, in our case, limiting role.

The actual growth setup corresponds to the PVT of AlN and is based on inductive heating of a graphite rf-heated susceptor with a TaC crucible inside. The scandium was added by placing scandium chunks in the already purified AlN source material [10]. Using this method, ScAlN mixed crystals (Fig. 5) were grown at different seed temperatures and ScN portions in the AlN source material. The growth rate was comparable to that obtained for AlN bulk crystals (around 100 µm/h).

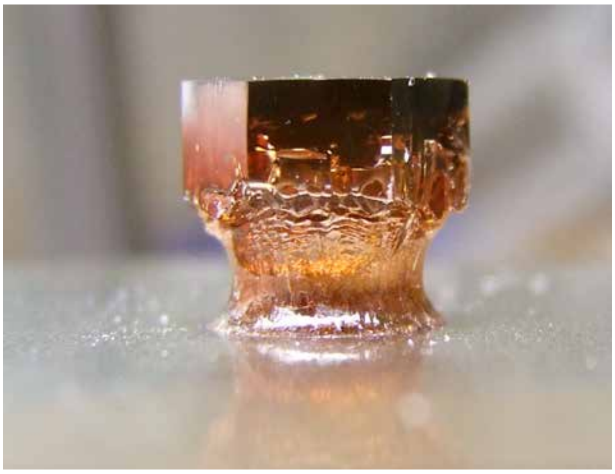

Fig. 5

AlScN single crystal (Ø~8mm, 0.4 at% Sc), growth interface above.

The scandium content was calculated on the basis of data from Moram et al. [5] and Vegard's law by high-precision x-ray measurement of the lattice parameters (Fig. 6). Despite the significant differences in ionic radii (Al^{3+}= 57 pm, Sc^{3+}= 83 pm), the lattice parameter change is only slightly beyond the detection limit due to the very low scandium content. A lower temperature is found to result in lower scandium content and vice versa, but the scandium content is about 15 times smaller than calculated from thermodynamic considerations.

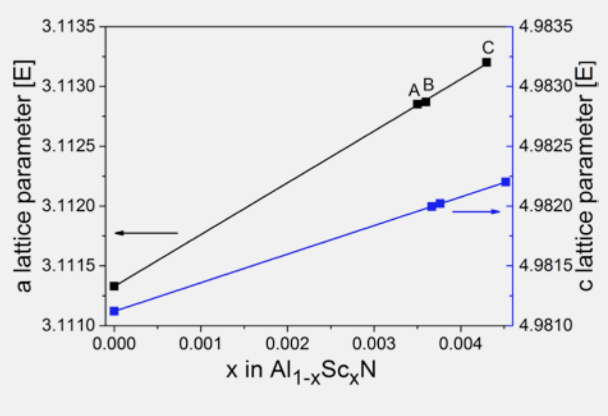

Fig. 6

High-precision x-ray measurement of the lattice parameters a and c of AlN and $Al_{1-x}Sc_xN$ single crystals grown at different seed temperatures (A - 2100°C, B - 2150°C, C - 2250°C). The figure is reprinted [10] with permission from Elsevier.

The thermodynamic considerations do not describe the real incorporation rates of scandium in AlN. The reason could be found in surface energies and residual impurities like oxygen and carbon as well as their volatile compounds with the crucible material that influence evaporation and condensation kinetics in PVT growth. Unfortunately, due to the very high process temperatures, it is hardly possible to measure these parameters and their influence on ScAlN growth.

In another series of growth experiments, the portion of scandium in the AlN source material was varied in a wide range, while the seed temperature T_S was kept constant at 2150°C and all other experimental parameters remained unchanged. Results are shown in Fig. 7.

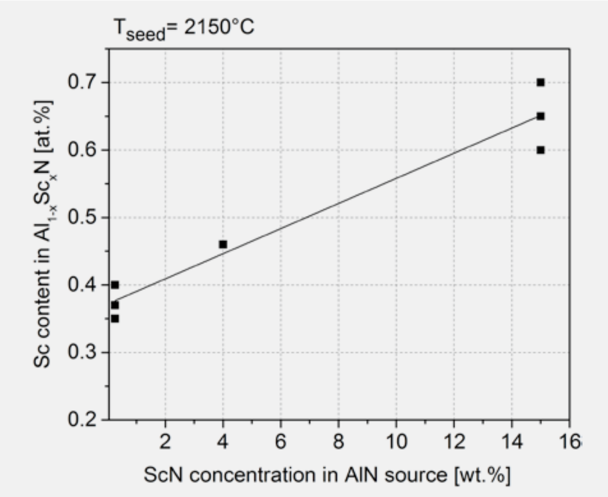

Fig. 7

Scandium concentration in $Al_{1-x}Sc_xN$ crystals grown at T_S = 2150°C as dependent on different ScN portions of the source AlN material. The figure is reprinted [10] with permission from Elsevier.

An increase of the scandium amount in the source material to 15 wt% led to a doubling of scandium incorporation. On the other hand, in all experiments, the ScN in the source was not completely consumed, and remains were observed, even with the smallest initial amount of 200 mg scandium in the source (with about 60 g of AlN). Also, XRF line scans of scandium along axial cuts of $Al_{1-x}Sc_xN$ crystals have revealed a continuous increase of the scandium content along the growth direction. That means, the scandium concentration increases with process time independently of the applied scandium amount (Fig. 8). A kinetic inhibition of ScN evaporation is therefore probable.

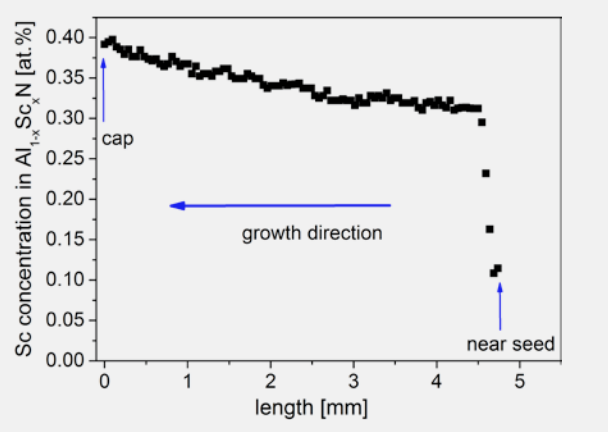

Fig. 8

Axial scandium incorporation during $Al_{1-x}Sc_xN$ crystals growth at T_S = 2150°C (XRF line scan). The figure is reprinted [10] with permission from Elsevier.

A second explanation would be that the equilibrium concentrations of aluminum and scandium species are present in the gas phase, but the scandium sticking (or segregation) coefficient is low in comparison to the aluminum one, as its ionic radius is too large to be incorporated in large amounts. The scandium atoms are thus accumulated at the growth interface, and as a consequence, the concentration of scandium incorporated in the crystal grows as well. It is evident that under the present conditions, no further increase in the scandium concentration in the ScAlN single crystals will be possible. However, a technological solution could be to keep the Al partial pressure in the growth chamber below the thermodynamic equilibrium value by means of diffusion barriers.

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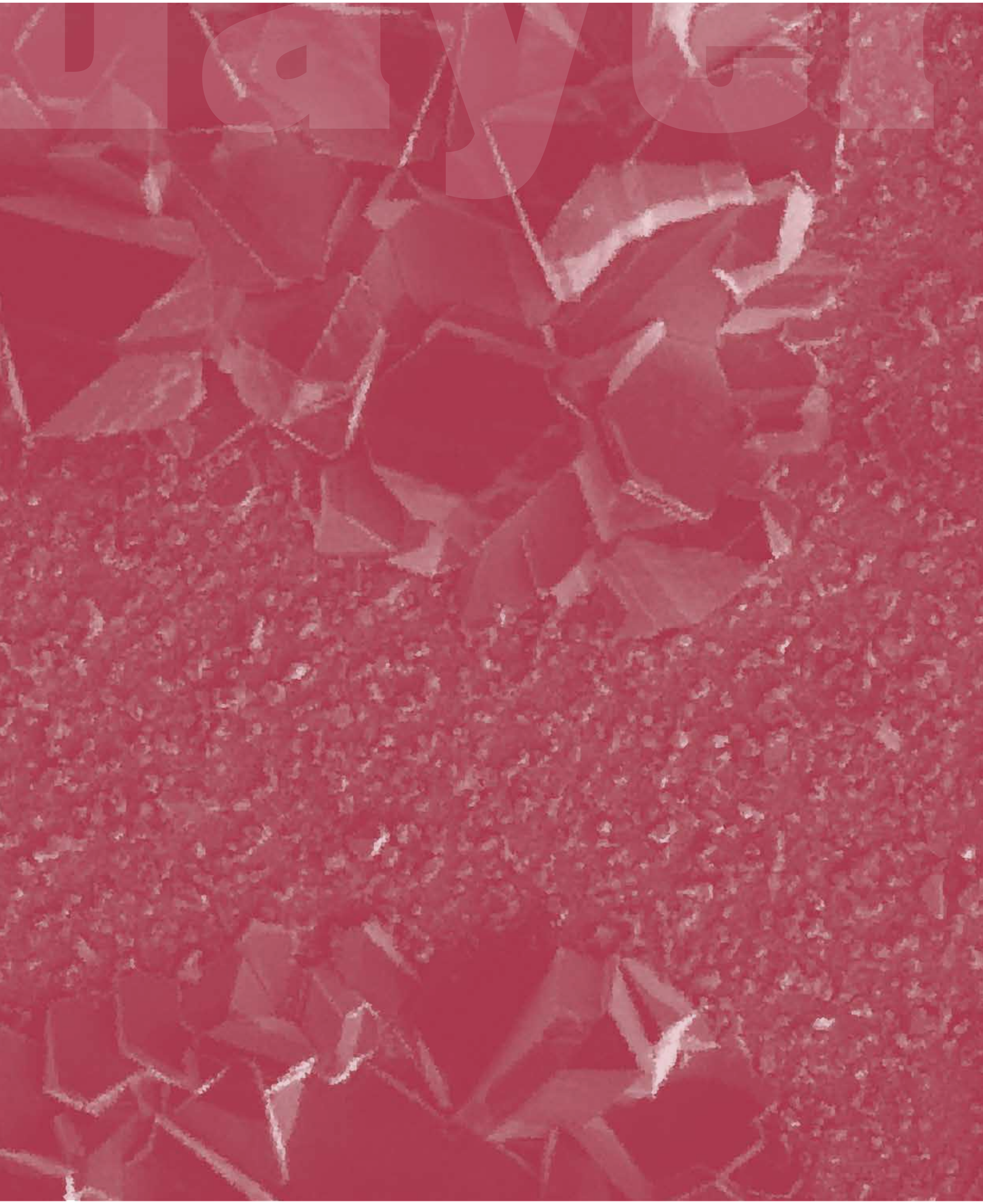
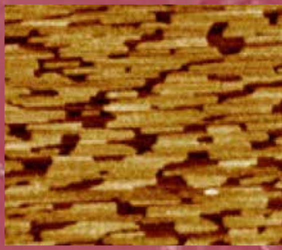
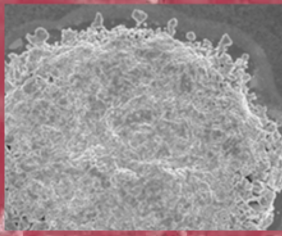
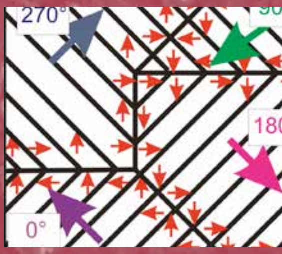
Schichten & Nanostrukturen



Layers & Nanostructures







Schichten & Nanostrukturen

Acting head of department: Dr. Torsten Boeck

Die Abteilung konzentriert sich auf die folgenden Aktivitäten:

- *Wachstum und Charakterisierung von mono- und polykristallinen Schichten sowie dreidimensionalen Nanostrukturen von Halbleitern und Oxiden für Anwendungen in der Mikroelektronik, Sensorik, Datenspeicherung und Photovoltaik*
- *Entwicklung von Kristallzüchtungs- und Analysemethoden*
- *theoretische Vorhersage und Modellierung von Wachstumsprozessen*

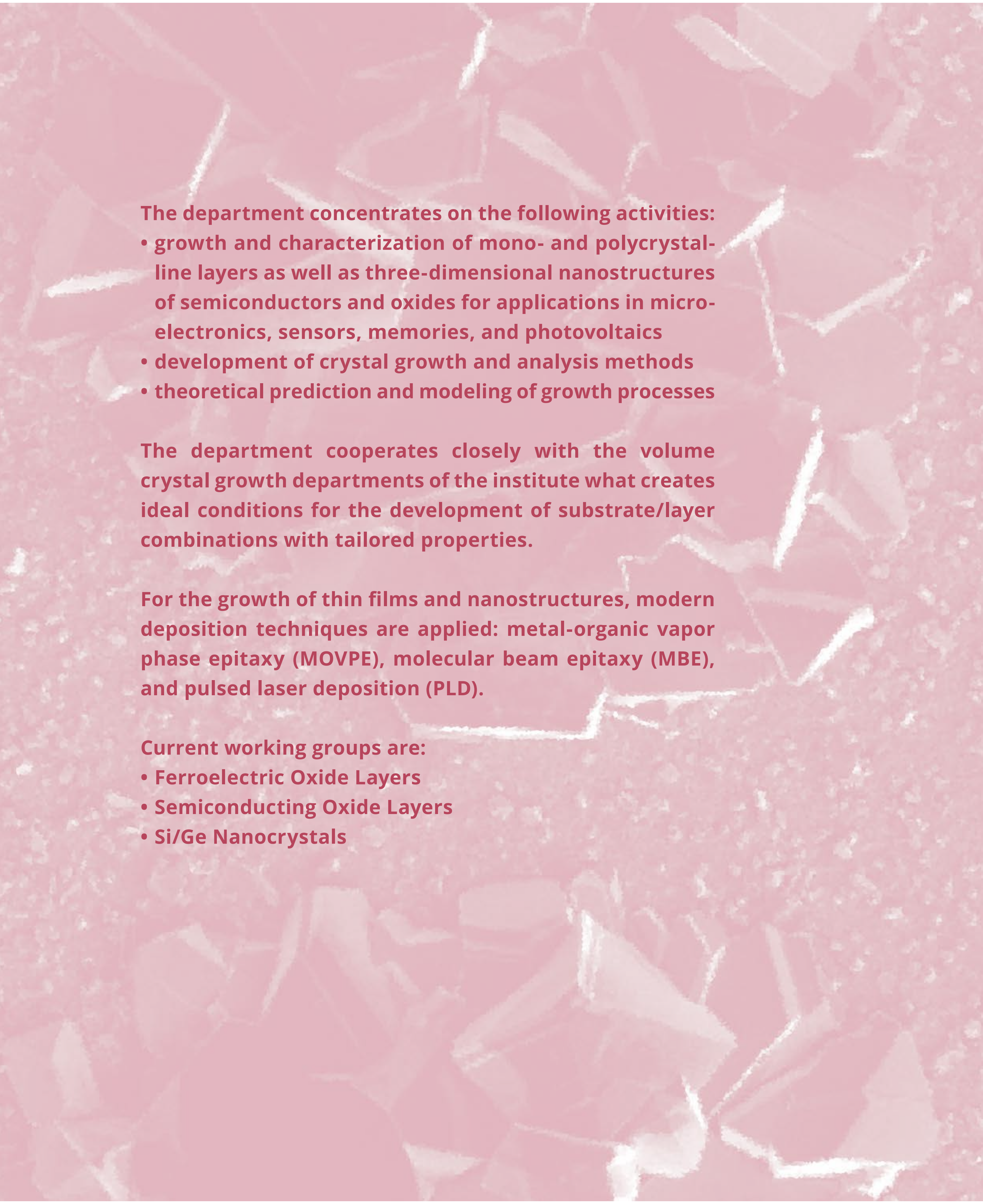
Die Abteilung arbeitet eng mit den Volumenkristallzuchtabteilungen des Instituts zusammen, was ideale Bedingungen für die Entwicklung von Substrat-Schicht-Kombinationen mit maßgeschneiderten Eigenschaften schafft.

Für das Wachstum von Dünnschichten und Nanostrukturen werden moderne Depositionstechniken eingesetzt: metallorganische Dampfphasenepitaxie (MOVPE), Molekularstrahlepitaxie (MBE) und gepulste Laserdeposition (PLD).

Aktuelle Arbeitsgruppen sind:

- *Ferroelektrische Oxidschichten*
- *Halbleitende Oxidschichten*
- *Si/Ge Nanostkristalle*

Layers & Nanostructures



The department concentrates on the following activities:

- growth and characterization of mono- and polycrystalline layers as well as three-dimensional nanostructures of semiconductors and oxides for applications in microelectronics, sensors, memories, and photovoltaics
- development of crystal growth and analysis methods
- theoretical prediction and modeling of growth processes

The department cooperates closely with the volume crystal growth departments of the institute what creates ideal conditions for the development of substrate/layer combinations with tailored properties.

For the growth of thin films and nanostructures, modern deposition techniques are applied: metal-organic vapor phase epitaxy (MOVPE), molecular beam epitaxy (MBE), and pulsed laser deposition (PLD).

Current working groups are:

- Ferroelectric Oxide Layers
- Semiconducting Oxide Layers
- Si/Ge Nanocrystals

Layers & Nanostructures:
Semiconducting Oxide Layers

Head Dr. Günter Wagner
Team Dr. M. Baldini, Dr. A. Popp, Dr. S. Bin Anooz, R. Grüneberg

Überblick

Galliumoxid (Ga_2O_3) hat in den letzten Jahren eine große Aufmerksamkeit als Material für Bauelemente in der Leistungselektronik erfahren. Aufgrund der Verfügbarkeit von großflächigen einkristallinen Wafern und der großen Bandlücke von etwa 4,8 eV bietet β-Ga_2O_3 ein großes Potenzial für elektronische Hochspannungsbauelemente, die die Leistung von Galliumnitrid (GaN) und Siliciumcarbid (SiC) mit Bandlücken von etwa 3,4 eV übertreffen könnten. Die Aktivitäten zur Züchtung von β-Ga_2O_3-Einkristallen, zur Abscheidung homoepitaktischer Dünnschichten und zur Entwicklung von elektronischen Bauelementen für die Leistungselektronik sind in den letzten zwei Jahren besonders stark gestiegen. In Japan und in den USA wurde die Forschung zu Ga_2O_3 durch neue umfangreiche Förderprogramme verstärkt.

Im Jahr 2017 setzte die Gruppe die sehr erfolgreichen Arbeiten zur Entwicklung homoepitaktischer Schichten durch Einsatz der metall-organischen Gasphasenepitaxie (MOVPE) fort. Die dafür eingesetzten (100) orientierten Ga_2O_3-Substrate wurden aus Einkristallen der Gruppe „Oxide & Fluoride“ präpariert, die nach der Czochralski-Methode gezüchtet wurden. Homoepitaktische Schichten, die auf 6° off-orientierten (100) β-Ga_2O_3-Substraten aufgewachsen sind, zeigten eine geringe Dichte an planaren Defekten und eine freie Trägermobilität von bis zu 125 cm^2/Vs bei einer Trägerkonzentration von 2x10^{17} cm^{-3}. Der n-Typ-Dotierungsbereich konnte zwischen 1x10^{17} cm^{-3} und 5x10^{19} cm^{-3} reproduzierbar eingestellt werden. Vergleichende MOVPE-Experimente wurden auf (010) orientierten Ga_2O_3-Substraten durchgeführt, die in Japan (TAMURA) mit den EFG-Verfahren gezüchtet wurden. Die strukturellen und elektronischen Eigenschaften von Schichten, die auf beiden Substrattypen gewachsen sind, sind ähnlich. Sie wurden in Zusammenarbeit mit den Gruppen „Elektronenmikroskopie“ und „Physikalische Charakterisierung“ einschließlich elektrischer und optischer Charakterisierung sowie Röntgendiffraktionsanalyse (XRD) analysiert und interpretiert.

Die Zusammenarbeit mit dem Ferdinand-Braun-Institut, Leibniz-Institut für Höchstfrequenztechnik, Berlin, und dem Air-Force Research Laboratory, USA, zur Bewertung der MOVPE-gewachsenen Schichten in einem Bauelemente-bezogenen technologischen Prozess wurde fortgesetzt. Erste Demonstrator-Bauelemente, die das große Potential als Material für die Leistungselektronik belegen, wurden von den Kooperationspartnern präsentiert. Die wissenschaftlichen Ergebnisse unserer Forschung und der Zusammenarbeit mit den Kooperationspartnern wurden auf vier internationalen Konferenzen vorgestellt und in drei Veröffentlichungen publiziert [1, 2, 3].

In 2017 starteten drei Projekte, die sich auf die Abscheidung von homoepitakischen β-Ga_2O_3-Schichten konzentrieren: das Verbundprojekt „Oxitherm“ in Kooperation mit dem Fachbereich Physik der Humboldt-Universität zu Berlin; ein vom Air Force Office for Scientific Research, USA, gefördertes Projekt; und schließlich wurde im Oktober 2017 das Projekt „Oxikon“, ein Gemeinschaftsprojekt in Zusammenarbeit mit dem Ferdinand-Braun-Institut, Leibniz-Institut für Höchstfrequenztechnik und der Technischen Universität Berlin, im Rahmen des VIP+ Programms des BMBF gestartet.

Overview

β-Ga_2O_3 has recently received attention as the next generation of material for high-performance power electronics. The availability of large area single crystal substrates and the wide bandgap of 4.8 eV makes β-Ga_2O_3 the suitable candidate for high-voltage electronic devices that could potentially surpass the performance of GaN and SiC, which have more narrow bandgaps of 3.4 eV.

In the last two years, the activities related to the β-Ga_2O_3 bulk crystals growth, deposition of homoepitaxial thin films, and the engineering of devices for power electronic application have increased significantly. Japan and the USA governments have developed new extensive support programs to strengthen the research on Ga_2O_3.

In 2017, the IKZ continued the successful works in developing metal organic vapor phase epitaxy (MOVPE) growth of homo-epitaxial layers on β-Ga_2O_3 substrates. The substrates were prepared from single crystals grown by the “Oxides & Fluorides” group at the IKZ using the Czochralski method. Homoepitaxial layers grown on 6° off-oriented (100) β-Ga_2O_3 substrates exhibited low density of planar defects and free carrier mobility up to 125 cm^2/Vs at a carrier concentration of 2x10^{17} cm^{-3}. We also were able to tune reproducibly the n-type doping range between 1x10^{17} cm^{-3} und 5x10^{19} cm^{-3}. Comparative MOVPE experiments were performed on (010)-oriented Ga_2O_3 substrates grown in Japan (TAMURA) using edge-defined film-fed growth (EFG) methods.

The structural and electronic properties of layers grown on both types of substrates are similar. They were analyzed and interpreted in collaboration with the groups “Electron Microscopy” and “Physical Characterization” including electrical and optical characterization as well as X-ray diffraction (XRD) analysis.

We continued the collaboration with the Ferdinand-Braun-Institute, Leibniz-Institut für Höchstfrequenztechnik (FBH), Berlin and the Air-Force Research Lab (ARL), USA, to evaluate the MOVPE-grown layers in a device related technological process. The scientific results of our research and collaboration were presented at four international conferences and published in three papers [1, 2, 3].

In 2017, three projects focusing on the deposition of homoepitaxial β-Ga_2O_3 layers by MOVPE have started: The DFG-funded joint project “Oxitherm” in cooperation with the Department of Physics of Humboldt-Universität zu Berlin; one project supported by the Air Force Office for Scientific Research, USA, and, finally, “Oxikon” – a joint project in cooperation with Ferdinand-Braun-Institut, Leibniz-Institut für Höchstfrequenztechnik (FBH) and Technische Universität Berlin in the frame of the BMBF VIP+ program.

Results

Investigation of the surface diffusion process of gallium on the (100)-β-Ga_2O_3 surface

In the first half of 2017, surface diffusion process of gallium (Ga) atoms on the (100)-oriented β-Ga_2O_3 surface had been in the focus of the research. We performed a set of MOVPE experiments with short deposition time in the range 10 to 40 s and over the temperature range from 750 to 850 °C. The analysis of the corresponding surface morphologies by atomic force microscopy revealed the formation of thermodynamically stable nucleation seeds and two-dimensional islands. We have calculated the activation energy barrier for the diffusion of gallium on the surface by statistically analyzing the nucleation seeds in these experiments. On freshly cleaved substrates, the activation energy barrier for 10 s deposition at a temperature range from 750 °C to 850 °C is $E_d = 3.1 \pm 0.2$ eV. However, identical experiments on aged substrates (freshly cleaved substrates at contact with room atmosphere for 24h) showed a lower activation energy barrier of 1.3 ± 0.4 eV. We ascribe this lowering of the energy barrier to the adsorption of gaseous compounds contained in the room atmosphere (CO_2, O_2 or H_2) on the freshly cleaved surface. That would allow the formation of gallium suboxide compounds, which evaporate at elevated temperatures.

The dimensions and density of nucleation seeds depend on the deposition temperature and time. At higher temperatures, the diffusion length is longer and, therefore, large elongated two-dimensional islands form, but their density is low. On the opposite, at lower temperatures, the diffusion length is limited, which promotes the formation of smaller islands but with larger density. We have assigned the growth mechanism observed in these experiments to the *Frank-van der Merve* layer-by-layer growth model.

The substrate before deposition has, within the margin of errors, an atomically flat surface (Fig. 1 (a)). After 10 s of β-Ga_2O_3 deposition (Fig. 1 (b)), first nuclei become visible, and they transform into elongated two-dimensional islands after 15 s deposition (Fig. 1 (c)). The length-to-height aspect ratio of the islands is about 2 and the surface roughness value RMS increases up to 174 pm. After 20 s of deposition, the length and width of two-dimensional islands increase, but the aspect ratio remains the same. New nucleation seeds appear between the existing islands, and the islands start to coalesce (Fig. 1 (d)). After 25 s of deposition, the two-dimensional islands cover the entire surface of the substrate (Fig. 1 (e)), and the roughness reaches the highest value of RMS = 222 pm. Further increasing the time (30 s), we observe large coalesced areas (Fig. 1 (f)), and after 35 s of β-Ga_2O_3 deposition the first layer on the surface is nearly completed (Fig. 1 (g)). After 40 s, the closed layer β-Ga_2O_3 covers the whole surface of the substrate (Fig. 1 (h)). The thickness of the closed layer is about 600 pm, which corresponds to a half-height of the β-Ga_2O_3 unit cell.

Ga-diffusion on β-Ga_2O_3 surfaces has been investigated by Denis Meiling from the Technical University, Department of Chemistry, in a framework of his Master thesis [4] under the supervision of Dr. M. Albrecht and Dr. M. Baldini from the IKZ.

In the second half of the year, within the scope of two new projects, we have performed MOVPE experiments to grow epitaxial layers for the project partners at the Department of Physics of Humboldt-Universität zu Berlin, Ferdinand-Braun-Institut, Leibniz-Institut für Höchstfrequenztechnik (FBH) and the Air Force Office for Scientific Research, USA. Additionally, we have delivered epitaxial layers to the partners in the Leibniz GraFOx ScienceCampus, and to Prof. Schubert from the University of Nebraska (USA) and SMI Inc., New Jersey (USA) on the basis of bilateral scientific cooperation.

The goal was to obtain n-type β-Ga_2O_3 layers with thicknesses between 20 and 500 nm, free carrier concentration of $1x10^{18}$ cm^{-3}, and carrier mobility of about 80 cm^2/Vs. The layers were doped with silicon from tetraethyl orthosilicate as metalorganic source.

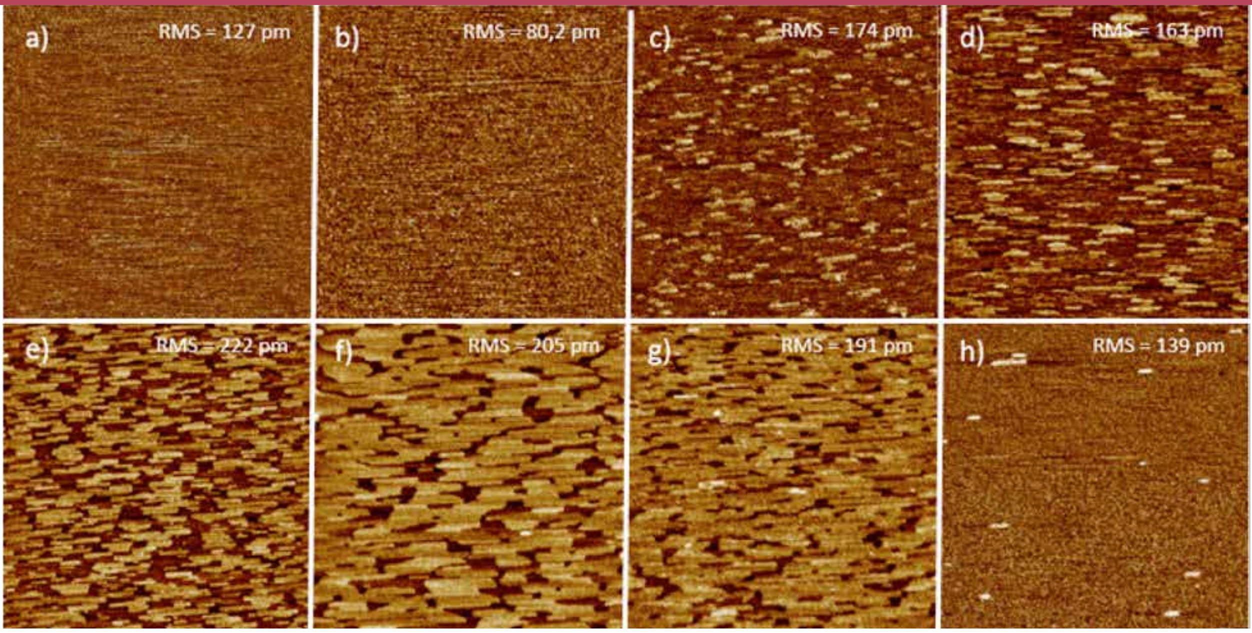


Fig 1
AFM pictures of β-Ga_2O_3 deposition series at 800°C:
a) pure substrate, b) 10 s, c) 15 s, d) 20 s, e) 25 s, f) 30 s,
g) 35 s and h) 40 s.
The size of the shown AFM scans are 500 x 500 nm.

We performed a number of experiments where the insulating β-Ga_2O_3 (010) substrate was overgrown with 200 nm n-type β-Ga_2O_3, (n_D-n_A: 3x10^{17} cm^2, μ about 110 cm^{-2}/Vs) and a 20 nm thick n-type β-Ga_2O_3 layer (n_D-n_A 5x10^{19} cm^{-3}, μ of about 60 cm^{-2}/Vs).

These layered structures served as the material basis for project partners to develop and produce field effect transistors prototypes. The characteristics of these demonstrators showed the high structural quality of the layers and thus the great potential of β-Ga_2O_3 for power electronics devices [2, 3, 5].

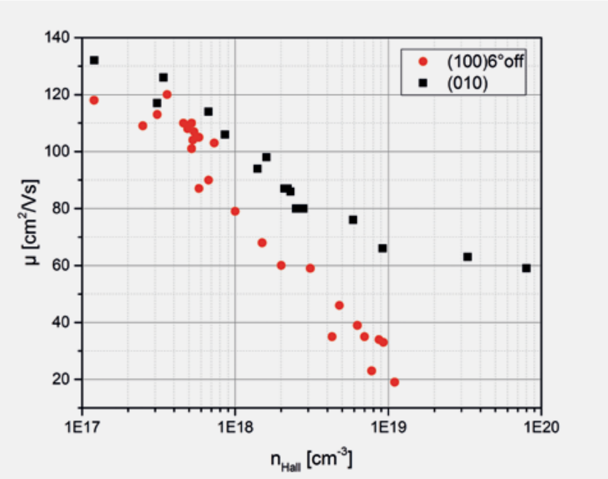


Fig. 2
Dependence of carrier mobility μ on free carrier concentration n in homo-epitaxial β-Ga_2O_3 layers grown by MOVPE.

In-situ Control of Ga_2O_3 Growth

At the end of the year, thanks to the funding of the EFRE project "Application Laboratory for Oxide Electronics", we were able to purchase optical in-situ process monitoring system from LayTec GmbH for our MOVPE facility (Fig. 3).

The optical detector of LayTec's EpiNet monitor combines measurements of temperature and reflectance at three different wavelengths (405, 633 and 951 nm). By measuring the reflectance, we can monitor the growth rate, film thickness, stoichiometry changes and morphology. This in-situ monitor allows to follow the growth process of the film step-by-step and accordingly adjust the growth conditions.

By changing the optical signals, we can use the in-situ monitor to observe an interruption during the growth process, to remove or decrease any memory effects, or to achieve sharp interfaces between layers.

To evaluate growth rate and film thickness, the refractive indexes for film and substrate should be different, which is not the case for homoepitaxial growth. Therefore, for these purposes, we used films grown on Al_2O_3 substrates during the same run.

Figure 4 (a) and (b) show the in-situ reflectance measurements at different deposition temperature and the evaluated growth rate, respectively. The growth rate was lower at higher temperatures, presumably, due to the formation and desorption of gallium suboxides [6].

The film thicknesses evaluated in-situ by reflectance monitoring and ex-situ by spectroscopic ellipsometry agree well.

In 2018, we plan to use the optical in-situ process monitoring system to grow delta-doped β-Ga_2O_3 layers, as well as β-$(In,Ga)_2O_3$ and β-$(Al,Ga)_2O_3$ thin films since in these systems the refractive index of the films differs from that of the substrate.

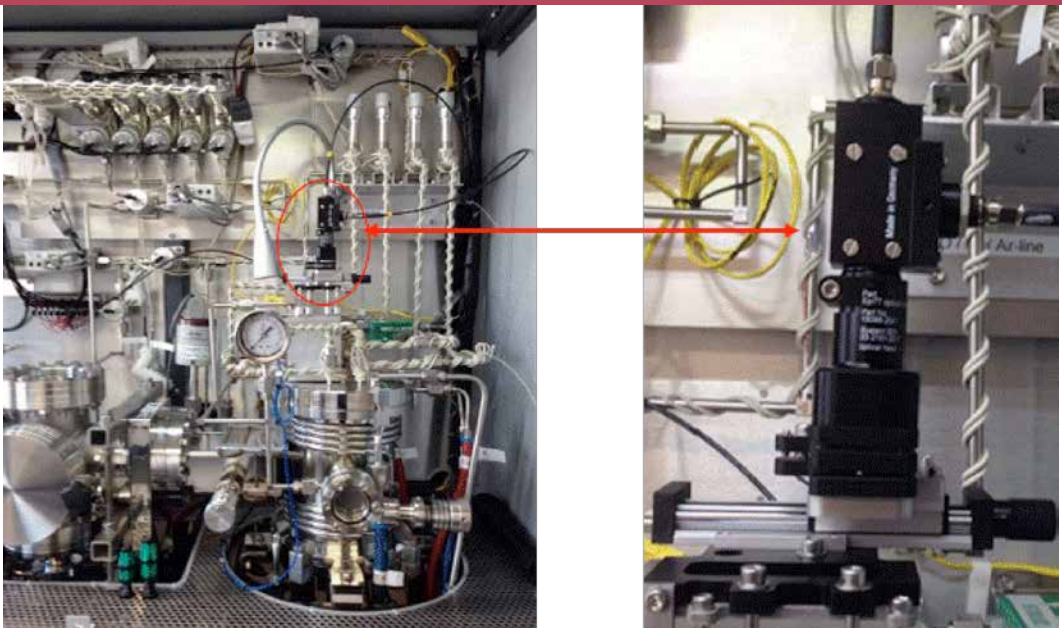


Fig. 3
EpiNet in-situ monitor installed on our MOVPE facility.

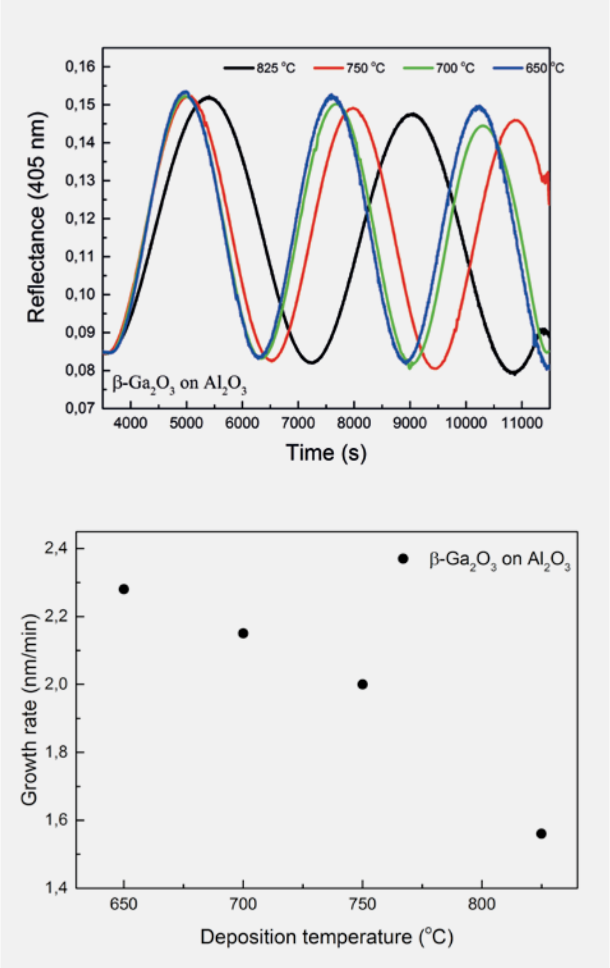


Fig. 4
a) Reflectance (wavelength 405 nm) versus growth time.
b) Growth rate versus deposition temperature for β-Ga_2O_3 thin films on Al_2O_3 substrates.

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Layers & Nanostructures:
Si/Ge Nanocrystals

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Überblick

Mikro- und nanoskalige kristalline Materialien für die effektive Energiewandlung stehen im Fokus der Forschungstätigkeit der Gruppe [1]. Die Arbeiten konzentrieren sich auf drei Themen:
(1) Züchtung von Siliziumschichten auf kostengünstigen Substraten, insbesondere auf Glas,
(2) $CuIn_xGa_{1-x}Se_2$ (CIGSe) Materialforschung als Basis für Mikrokonzentrator-Solarzellen,
(3) Züchtung von Si-, Ge- und Si_xGe_{1-x}-Nanodrähten für Thermoelektrika.

Zum überwiegenden Teil sind die Themen projektfinanziert und erfolgen in Zusammenarbeit mit einer Vielzahl von nationalen und internationalen Partnern. So fördert die EU die ersten beiden Forschungsthemen im Rahmen jeweils eines Arbeitspakets des Photovoltaik-Projektes CHEETAH. Das zweite Thema wird zusätzlich durch das DFG-Projekt „Lokal gewachsene $Cu(In,Ga)Se_2$-Mikroinseln für Konzentratorsolarzellen" gefördert. Das dritte Thema zur thermoelektrischen Energiewandlung ist in ein regionales Netzwerk eingebunden, bei dem acht Doktoranden aus verschiedenen Institutionen unter Federführung der Brandenburgischen Technischen Universität Cottbus–Senftenberg (BTU) im Rahmen der Graduiertenschule „Functional Materials and Film Systems for Efficient Energy Conversion" (FuSion) zusammenarbeiten.

Während beim EU-Projekt eher anwendungsorientierte Forschung im Vordergrund steht, zielt das DFG-Projekt auf die Aufklärung grundlegender Fragestellungen bei der Materialentwicklung und Charakterisierung und bildet einen guten Rahmen für Qualifizierungsarbeiten. Es ermöglicht die Anwendung von Forschungsergebnissen der Bundesanstalt für Materialforschung und -prüfung (BAM) auf dem Gebiet der Lasertechnik im IKZ zur ortsdefinierten Keimbildung beim Wachstum von CIGSe-Inseln, die dann am Helmholtz-Zentrum Berlin für Materialien und Energie (HZB) hinsichtlich photovoltaischer Kenngrößen charakterisiert wurden.

An den Forschungsarbeiten zu den genannten Themen waren im Berichtszeitraum insgesamt fünf Doktorandinnen und Doktoranden, sowie vier Bachelor- bzw. Masterstudenten beteiligt. Drei studentische Qualifikationsarbeiten, zwei zur elektrischen Isolation von CIGSe-Inseln und eine zur maskenbasierten Positionierung von Indiumtröpfchen, konnten erfolgreich abgeschlossen werden. Im Rahmen des DAAD-Programms „RISE Weltweit" unterstützte eine US-amerikanische Studentin der University of California, San Diego, die Arbeiten der Gruppe für drei Monate.

Overview

The focus of the research activities of the group lies in the development and growth of micro- and nano-crystalline materials for applications in the field of energy conversion. [1] The work can be divided into three topics:
(1) Growth of silicon layers on low-cost substrates, especially on glass;
(2) Research on $CuIn_xGa_{1-x}Se_2$ (CIGSe) materials suitable for microconcentrator solar cells;
(3) Growth of Si, Ge and Si_xGe_{1-x} nanowires for thermoelectric applications.

The research on all three topics is predominantly financed by third-party funds and carried out in cooperation with numerous national and international partners. The first two topics are supported by the EU within two work packages of the photovoltaics project CHEETAH. The second topic was additionally supported by the DFG project "Locally grown $Cu(In,Ga)Se_2$ micro-islands for concentrator solar cells". The thermoelectric energy conversion topic is embedded in a regional network within the graduate school "Functional Materials and Film Systems for Efficient Energy Conversion" (FuSion), in which eight doctoral students from different institutions cooperate under the general leadership of the BTU Cottbus/Senftenberg.

The EU-funded project primarily focuses on application-oriented research, the DFG granted project on the field of materials research for photovoltaics aims at the solution of fundamental questions in materials development and characterization. It also creates a favorable base for the qualification of young scientists. This provides the opportunity to use research results on the field of laser technology achieved at the Federal Institute for Materials Research and Testing (BAM) for the locally defined nucleation of CIGSe microcrystals at IKZ, which were characterized at the Helmholtz-Zentrum Berlin für Materialien und Energie (HZB) concerning photovoltaic parameters.

Within the reporting period, five doctoral students, as well as four bachelor and master students were involved in the research work on the above-mentioned topics. Three students completed their research projects related to $Cu(In,Ga)Se_2$ for micro-concentrator-solar cells. Two of them focused on the electrical insulation of $Cu(In,Ga)Se_2$ islands, while the third dealt with the mask-based local formation of indium islands on polycrystalline surfaces.

A student from the University of California, San Diego, USA supported our activities within the framework of the DAAD program "RISE Weltweit" for three months.

Results

Growth of silicon layers on cost-efficient substrates

We use Steady-State Liquid Phase Epitaxy (SSLPE) from a tin solution as a method of choice to obtain a crystalline material with high perfection and purity. In this method, a graphite crucible contains tin solution and a silicon feedstock on the bottom of it; the substrate is then placed upside-down on a tin solution. Due to the temperature gradient, silicon dissolves from the feedstock, travels through the tin solution, and then recrystallizes at the colder substrate. This technique allows to perform a continuous growth instead of a batch-type process, and thus to avoid heating and cooling of the system, as well as replacement of consumables from run to run.

Two central research approaches exist to grow thin silicon layers on cost-efficient substrates for photovoltaics. In the first approach, funded by the EU CHEETAH project, detachable silicon layers [2, 3] are grown on reorganized porous silicon substrates provided by the project partner IMEC, Belgium. The preparation of such substrates includes porosification of Si(111) wafers by lithography and reorganization in a hydrogen atmosphere. This process results in a thin foil connected to the bulk substrate by multiple small silicon pillars. Such a structure is stable enough for the SSLPE, and the foil with the grown layer can be subsequently detached for solar cell processing, while the remaining substrate can be reused. Within the framework of the project, we demonstrated 20 – 70 µm thick Si epilayers that exhibit material properties suitable for solar cell production (see Figure 1).

In the second approach, we use borosilicate glass substrates covered with an amorphous Si (a-Si) seed layer. Our multi-chamber treatment suffers from the formation of a native oxide on the a-Si layer. To remove that oxide before the epitaxy, we applied a UV-laser treatment instead of a hardly controllable melt-back step. During this treatment, the a-Si surface is heated by laser pulses, which remove the native oxide but prevent surface melting. By proper adjustment of the scan distance of the single laser pulses, the oxides layer can be removed entirely, thus allowing adjacent silicon crystallite layers to grow on glass substrates (Figure 1 (right)).

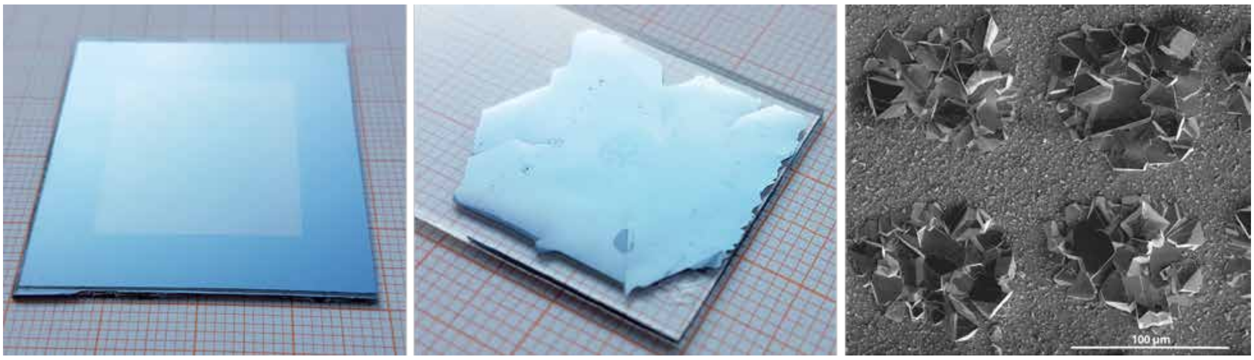
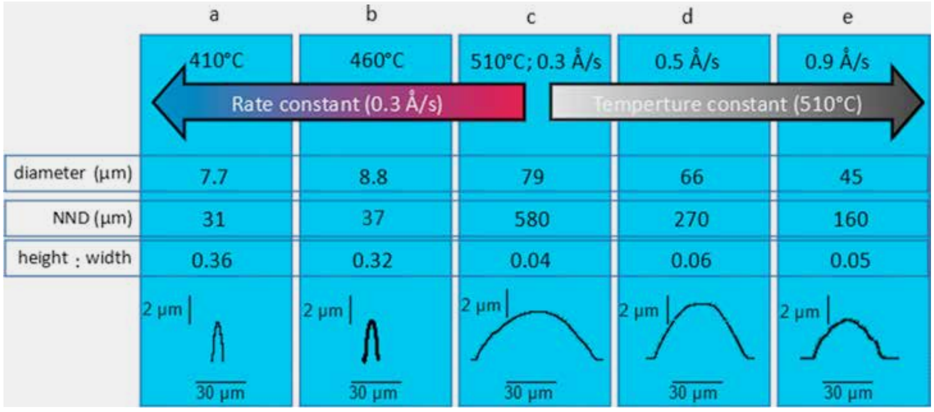


Fig. 1

Photograph of a reorganized porous Si substrate before epitaxy by SSLPE (left); Photograph of a detached epilayer (middle); SEM-image of the silicon crystallites grown on a-Si seed layer after the removal of the native oxide by four single UV-laser pulses (right).



	a	b	c	d	e
	410°C	460°C	510°C; 0.3 Å/s	0.5 Å/s	0.9 Å/s
diameter (µm)	7.7	8.8	79	66	45
NND (µm)	31	37	580	270	160
height : width	0.36	0.32	0.04	0.06	0.05

Fig. 2
Influence of substrate temperature (T) and deposition rate (r) on the morphology of indium islands. Scanning force microscopy [(a), (b) and profilometric (c), (d), (e)] cross sections are plotted with equal scales.

CIGSe microconcentrator solar cells

Cu(In,Ga)Se_2 (CIGSe) is a I-III-VI-compound semiconductor, used as a direct bandgap absorber material for photovoltaic energy conversion with an average efficiency of 22.9 % [4]. The volatile prices and limited resources for indium and gallium require material-saving concepts. Our bottom-up process to prepare micrometer-sized CIGSe islands at predefined sites on a molybdenum-covered glass substrate requires only 1 % of the material used for thin films. Subsequent deposition of gallium, copper, and selenium, as well as a two-stage annealing process, are extensively investigated to convert the raw deposit to CIGSe micro-absorbers with chalcopyrite structure.

Besides structuring of the substrate by the femtosecond laser [5] carried out in cooperation with the Bundesanstalt für Materialforschung und -prüfung (BAM), we also developed a mask-based approach that enables the positioning of CIGSe islands at predefined sites. We were able to control island size, areal density, and aspect ratio to achieve the desired array dimensions by adjusting the indium deposition rate and the substrate temperature [6] (see Figure 2 for details).

In case of CISe islands a careful adjustment of the copper to indium ratio while maintaining a slight excess of copper led to a homogeneous elemental distribution that is characteristic for a pure CISe phase. The resulting CISe micro-absorbers are measured under standard illumination conditions (1 sun) at the *Helmholtz Zentrum Berlin für Materialien und Energie* (HZB). They exhibit an optical band gap energy of about 1.03 eV and high spatial homogeneity of photoluminescence across the entire islands. The test devices offer a diode behavior and have a solar efficiency of (2.9 ± 0.2) %. [7]

The research was mainly focused on replacing indium with gallium. CIGSe islands were prepared by improving the gallium deposition and the selenization temperature. Due to the very different melting points of indium and gallium, we prepared In-Ga micro-islands in a sequential process: starting with In and continuing with Ga, which accumulates circularly around the indium island (Fig. 3 (a)). We influenced the mixing of the components within the CIGSe islands by changing the temperature in the second heating step of selenization. Samples produced with high selenization temperatures (540 °C) show promising results regarding homogeneity (Fig. 3 (c)) and band gap (Fig. 3 (d)) of the resulting CIGSe micro-absorbers (Fig. 3 (b)). The structural and optoelectronic properties of these absorbers are comparable to those commonly reported for CIGSe films. Characterization of the resulting devices under a solar simulator yielded an efficiency of 3.36 % at 20 suns, which corresponds to a relative increase of 138 % compared to 1 sun. [8]

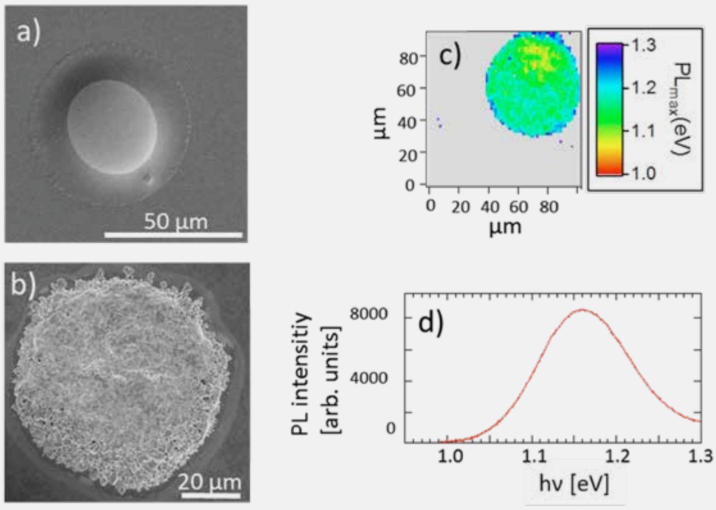


Fig. 3
SEM images of a) In-Ga-island and b) CIGSe island. c) Photoluminescence (PL) map of the peak emission energy, for a CIGSe island selenized at 540 °C. d) Averaged spectra of c) as a function of energy.

SiGe nanowires for thermoelectric applications

In our group, we investigate the growth of Si_xGe_{1-x} nanowires by molecular beam epitaxy (MBE) in ultra-high vacuum. Si/Ge nanowires with small diameters (about several tenth nanometers) have reduced thermal conductivity, and thus are promising for thermoelectric devices in the medium temperature range. In nanowires, the higher temperature gradient can be achieved as compared to the bulk material, which results in a higher output voltage and therefore an improved efficiency of the device.

The Si/Ge nanowire growth process is based on the vapor-liquid-solid (VLS) mechanism, where liquid Au droplets catalyze the nanowire formation. During the deposition of Au onto the Si substrate, eutectic Si/Au droplets form on the surface. These act as the nucleation center for the nanowire growth during deposition of Si/Ge material from the MBE evaporation sources.

When using bare Si substrates, we observe a stochastic distribution of Au droplets with varying diameters. It results in non-uniform lengths of the resulting nanowires as well as the unwanted deposition of active material in the interspace between the nanowires. The dependence of droplet size and distribution from Au deposition rate and the substrate temperature was carefully investigated.

To tackle these challenges, modified Si substrates [9] from Leibniz Institute for High-Performance Microelectronics (IHP) are under investigation. These substrates possess regularly arranged circular Si pillars with diameters in the range of 30 – 50 nm protruding from an 800 nm thick SiO_2 layer. First of all, the deposition process was adjusted so that Au exclusively nucleates on the crystalline Si pillars, to avoid the epitaxial crystallization of Si and Ge on the oxide. Recent results show the successful deposition of Au and nucleation of Au droplets on the patterned Si(100) IHP substrates (cf. Fig. 4).

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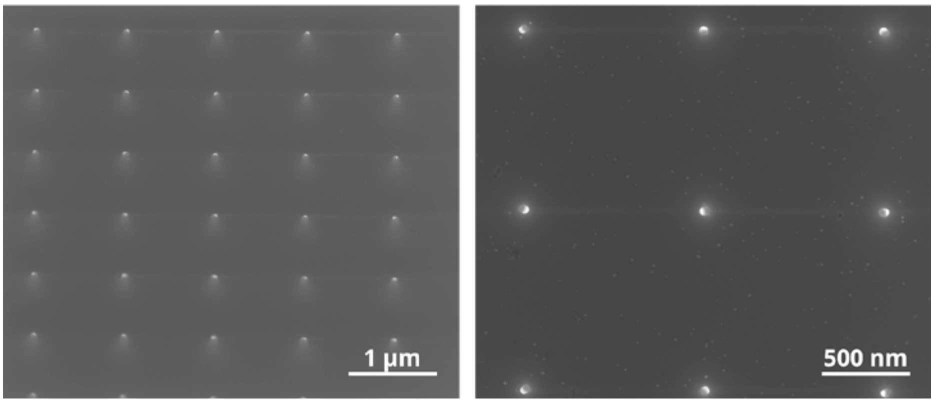

Fig. 4
(left) 45° tilted SEM image of patterned Si(100) sample with circular crystalline Si openings between SiO_2 matrix, where openings have a distance of 1 µm. Conical shape below the bright spots is the Si pillar structure beneath the SiO_2. (right) Top view SEM image of the same sample with higher magnification. Bright spots on top of Si openings correspond to Au, which was also confirmed by EDX analysis. Several spots at the darker SiO_2 matrix indicate the nucleation of Au on top of the oxide.

Layers & Nanostructures:

Ferroelectric Oxide Layers

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Überblick

Perowskite mit der allgemeinen Formel ABO_3 stellen eine umfangreiche Klasse von Funktionsmaterialien dar, deren Eigenschaften entscheidend von ihrer exakten chemischen Zusammensetzung, den Gitterverzerrungen (z.B. durch Einbringen von Gitterverspannungen) und Punktdefekten abhängen. $SrTiO_3$ ist ein Prototyp der Perowskite, der viele spannende und vielseitige Eigenschaften, wie eine hohe Dielektrizitätskonstante und bipolares resistives Schaltverhalten, bietet. Obwohl $SrTiO_3$ schon vielfach untersucht wurde, gibt es noch viele offene Fragen. Ein Teilprojekt des Leibniz WissenschaftsCampus GraFOx (Growth and fundamentals of oxides for electronic applications) zielt darauf ab, $SrTiO_3$ als Modellsystem für eine grundlegende Untersuchung von Defekten und deren Einfluss auf die funktionellen Eigenschaften von Perowskiten wie Ferroelektrizität oder elektrische Leitfähigkeit zu nutzen. Zu diesem Zweck haben wir Wachstumsexperimente mit gepulster Laserabscheidung (PLD) begonnen und werden diese in 2018 mit der metallorganischen Gasphasenepitaxie (MOVPE) fortführen. Aufgrund des Zusammenspiels von strukturellen, chemischen und elektrischen Eigenschaften wurden diese Untersuchungen in enger Zusammenarbeit mit den IKZ Gruppen „Elektronenmikroskopie“ und „Physikalische Charakterisierung“ sowie mit Dr. M. Ramsteiner vom Paul-Drude-Institut für Festkörperelektronik (PDI) durchgeführt.

Unsere Gruppe arbeitet seit mehreren Jahren an der Perowskitstruktur $K_xNa_{1-x}NbO_3$ mit dem Ziel, Schichten mit hervorragenden ferro-/piezoelektrischen Eigenschaften zu entwickeln. Dieses Material wird oft als bleifreie Alternative zu Oxiden wie $Pb(Zr,Ti)O_3$ für Dünnschichtsensoren, Speicherzellenkondensatoren oder für die Energieerzeugung (Energy Harvesting) angesehen. Darüber hinaus bietet es hervorragende Kopplungskoeffizienten für z.B. akustische Oberflächenwellenbauelemente. Im IKZ werden diese Schichten mit der PLD und MOVPE Technik gezüchtet. Bisher ist das IKZ weltweit das einzige Institut, das in der Lage ist, stöchiometrische $K_xNa_{1-x}NbO_3$ Dünnschichten unterschiedlicher Stöchiometrie mittels MOVPE zu züchten.

Zur Erweiterung der Materialbasis und der Charakterisierungsexpertise wurde 2017 das EFRE-Projekt „Applikationslabor für Materialien der Oxidelektronik“ eingeworben. In diesem Rahmen haben wir einen neuen MOVPE Aufbau beschafft. Die Installation der Anlage erfolgt im Frühjahr 2018. Unser zentraler Ansatz zur Einstellung der Materialeigenschaften ist der präzise Einbau einer Gitterverspannung in dünnen Schichten durch das epitaktische Wachstum der Schichten auf gitterfehlanpassenden Oxidsubstraten. In Zusammenarbeit mit den Gruppen „Physikalische Charakterisierung“ und „Elektronenmikroskopie“ ermitteln wir die strukturellen Eigenschaften der Filme, wie Orientierung und Symmetrie, und analysieren, wie diese mit den funktionellen Eigenschaften korrelieren. Im Hinblick auf die Verwendung von $K_xNa_{1x}NbO_3$ Dünnschichten in akustischen Oberflächenwellenbauelementen arbeiten wir im Rahmen des DFG Projektes mit der AG Prof. Dr. R. Wördenweber am Forschungszentrum Jülich zusammen.

Vor einem Jahr haben wir damit begonnen, unsere Materialbasis um Nioboxide zu erweitern. Das im Rahmen des Leibniz-Wettbewerbs „Physik und Kontrolle von Defekten in Oxidschichten für die adaptive Elektronik“ geförderte Projekt will Nb-Oxid Dünnschichten für resistive Schaltelemente oder Selektoren nutzen. Seit März 2017 werden NbO_2 und Nb_2O_5 Schichten auf verschiedenen Substraten epitaktisch aufgewachsen, um deren elektrische und optische Eigenschaften zu bestimmen.

Im Jahr 2017 warb das IKZ erfolgreich das Leibniz-Wettbewerbsprojekt „Barium-Stannat-basierte Heterostrukturen für elektronische Anwendungen - Bastet“ ein. Das Projekt ist eine Kooperation von drei IKZ-Gruppen („Oxide & Fluoride“, „Physikalische Charakterisierung“, „Ferroelektrische Oxidschichten“), einer PDI-Gruppe (Dr. O. Bierwagen), der TU Berlin (Prof. Dr. H. Eisele) und der HU Berlin (Prof. Dr. C. Draxl) und wird für drei Jahre gefördert. Im Rahmen dieses Projektes werden die funktionellen Eigenschaften von $K_xNa_{1-x}NbO_3$ Dünnschichten grundlegend untersucht mit dem Ziel, deren (ferro-)elektrische Eigenschaften wesentlich zu verbessern.

Im Jahr 2017 schloss Dr. Dorothee Braun ihre Promotion „Strain-phase relations in lead-free ferroelectric $K_xNa_{1-x}NbO_3$ epitaxial films for domain engineering“ mit Auszeichnung (summa cum laude) ab.

Overview

Perovskites with the general formula ABO_3 represent a wide class of functional materials whose properties crucially depend on their exact chemical composition, lattice distortions (caused, e.g., by the incorporation of lattice strain) and point defects. $SrTiO_3$ is a prototype of the perovskites, which offers many exciting and versatile properties like high dielectric constant and bipolar resistive switching behavior. Although $SrTiO_3$ is often investigated, there are still many open questions. A subproject within the Leibniz ScienceCampus GraFOx (Growth and fundamentals of oxides for electronic applications) aims to use $SrTiO_3$ as a model system for a basic study of defects and their impact on functional properties of perovskites like ferroelectricity or electrical conductivity. For this purpose, we have started growth experiments with pulsed laser deposition (PLD) and will continue with metal-organic vapor phase epitaxy (MOVPE) in 2018. Due to the interplay between structural, chemical and electrical properties, these investigations have been performed in close cooperation with the IKZ groups "Electron Microscopy" and "Physical Characterization" as well as with Dr. M. Ramsteiner from the Paul-Drude-Institut für Festkörperelektronik (PDI).

Our group has now been working for several years on the perovskite material $K_xNa_{1-x}NbO_3$ aiming to develop films with excellent ferro-/piezoelectric properties. This material is often considered as a lead-free alternative to oxides like $Pb(Zr,Ti)O_3$ for thin-film sensors, single memory cell capacitors or energy harvesting applications. Furthermore, it provides excellent coupling coefficients for, e.g., surface acoustic wave devices. In IKZ, these films are grown by PLD and MOVPE techniques. Up to now, IKZ is the only institute worldwide that is able to grow low defect, stoichiometric $K_xNa_{1-x}NbO_3$ thin films with different stoichiometry by MOVPE.

To expand the material basis and characterization expertise, the EFRE project "Applikationslabor für Materialien der Oxidelektronik" (Application Laboratory for Oxide Electronic Materials) was acquired in 2017. Within its framework, we obtained a new MOVPE set-up. The installation of the set-up will take place in spring 2018. Our essential approach to adjust material properties is to precisely introduce lattice strain into thin layers by epitaxy on lattice-mismatched oxide substrates. In cooperation with the group "Physical Characterization" and "Electron Microscopy", we determine structural properties of the films, like orientation and symmetry, and analyze how they correlate to the functional features. On the application of $K_xNa_{1-x}NbO_3$ thin films in surface acoustic wave devices, we collaborate with AG Prof. Dr. R. Wördenweber at Forschungszentrum Jülich through the funded DFG project.

One year ago, we started to broaden our material base to niobium oxides. The project funded in the frame of the Leibniz Competition "Physics and control of defects in oxide films for adaptive electronics" intends to exploit Nb-oxide thin films for resistive switching elements or selectors. Since March 2017, NbO_2 and Nb_2O_5 films have been epitaxially grown on different substrates to determine their electrical and optical properties.

In 2017, IKZ successfully acquired the Leibniz Competition project "Barium-stannate based heterostructures for electronic applications – Bastet". The project is a joint cooperation between three IKZ groups ("Oxides & Fluorides", "Physical Characterization", "Ferroelectric Oxide Films"), one PDI group (Dr. O. Bierwagen), the TU Berlin (Prof. Dr. H. Eisele) and the HU Berlin (Prof. Dr. C. Draxl) and provides funding for three years. In the framework of this project, the functional properties of $K_xNa_{1-x}NbO_3$ thin films will be fundamentally investigated with the aim to improve their (ferro-)electric properties substantially.

In 2017, Dr. Dorothee Braun completed her doctorate "Strain-phase relations in lead-free ferroelectric $K_xNa_{1-x}NbO_3$ epitaxial films for domain engineering" with the mark summa cum laude.

Results

Strained, ferroelectric $K_xNa_{1-x}NbO_3$ thin films

$K_xNa_{1-x}NbO_3$ thin films have been grown by MOVPE under different strain conditions by varying the potassium to sodium ratio ($0.60 < x < 0.80$) and using rare-earth scandate substrates ($TbScO_3$, $GdScO_3$, $SmScO_3$) with different in-plane lattice parameters. In case of $K_{0.7}Na_{0.3}NbO_3$ on $TbScO_3$, (average strain -1%) the periodically arranged ferroelectric stripe domains (Fig. 1(a)) can be described by $(001)_{pc}$ oriented M_C domains with monoclinic *Pm* symmetry (Fig. 1 (c)) with the lateral polarization component aligned along the main crystallographic axis of the substrate (Fig. 1 (b)). Moreover, upon decreasing compressive strain (by using $GdScO_3$ and $SmScO_3$ substrates), the stripe domains are similar (Fig. 1 (d)), but additionally, we observe the formation of a monoclinic M_A phase with *Cm* symmetry (Fig. 1 (f)). We attribute this to the fact that the strain state in these films approaches the M_C-M_A phase boundary in the strain phase diagram [1]. M_A domains also form a stripe domain pattern whereby the lateral polarization components are now rotated by 45° and oriented along $[11\text{-}2]_o$ or $[1\text{-}1\text{-}2]_o$ directions of the rare-earth scandate substrate (Fig. 1 (e)).

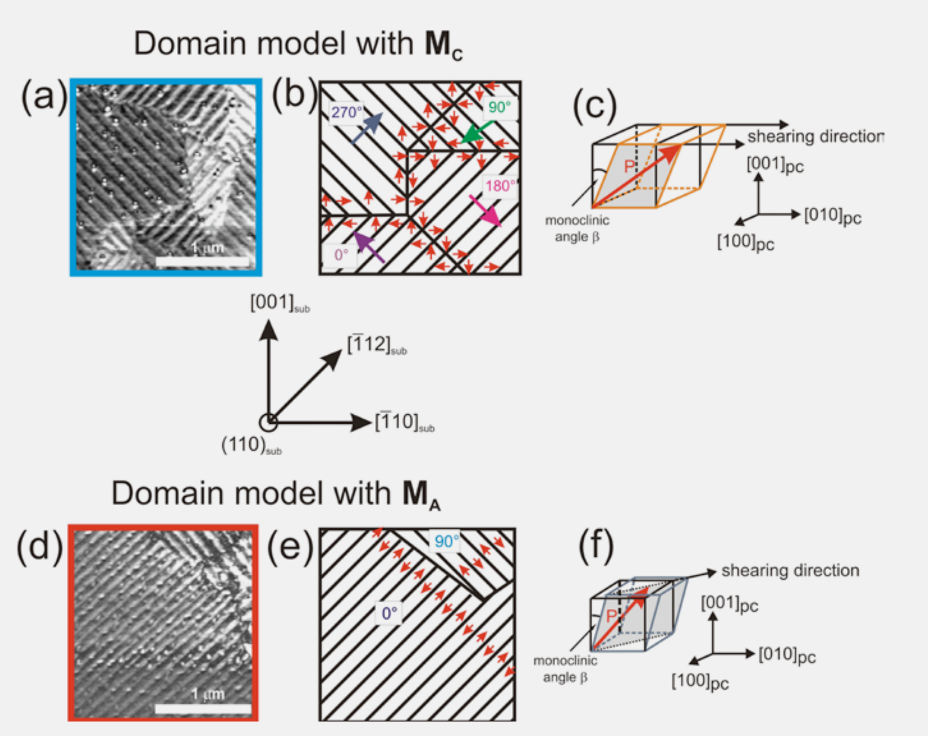

Fig. 1
(a) Lateral piezoresponse force micrograph (LPFM) of the domain pattern of $K_{0.7}Na_{0.3}NbO_3$/$TbScO_3$; (b) model with M_C domains; (c) unit cell with Pm symmetry; (d) LPFM of the domain pattern of $K_{0.7}Na_{0.3}NbO_3$/$GdScO_3$; (e) model with M_A domains; (f) unit cell with Cm symmetry.

These results are of particular interest for applications in surface acoustic wave devices. In collaboration with the group of R. Wördenweber from Forschungszentrum Jülich, we have shown that the propagation of surface acoustic waves in thin films grown on $TbScO_3$ with *Pm* symmetry occurs selectively along the main crystallographic axis $[001]_o$ and $[1\text{-}10]_o$, while the films on $GdScO_3$ and $SmScO_3$ additionally show signals along the orthorhombic $[1\text{-}12]_o$ direction of the substrate. Besides the fact that the surface acoustic waves propagate along the direction that coincides with the in-plane orientation of the polarization vector of M_C and M_A domains, we should also emphasize that the obtained signal strength of up to 3.6 dB is remarkably high for 30 nm thin layers. In the next step, we will test the sensitivity of the structures for biosensor applications.

For monoclinic $K_{0.9}Na_{0.1}NbO_3$ thin films epitaxially grown on $NdScO_3$ substrates, we have investigated the formation process of a ferroelectric multi-rank domain pattern in the thickness range of 7 - 52 nm. For this film-substrate combination, the elastic strain energy density is degenerated for the two pseudocubic $(100)_{pc}$ and $(001)_{pc}$ orientations. For very thin films we observe an exclusive in-plane a_1a_2 stripe domain pattern. The retarded onset of the ferroelectric M_C phase at larger film thickness is accompanied by a thickness dependent transformation from stripe domains to a herringbone pattern and, eventually, to a checkerboard-like structure for the thickest film. From piezoresponse force microscopy (PFM) and x-ray diffraction (XRD) data, we could correlate the transformations in the domain arrangement and width to energetic aspects like depolarization field and anisotropic strain relaxation in the film. Furthermore, we have observed plastic strain relaxation for the M_C domains, while the a_1a_2 domains show a two-step strain relaxation mechanism. This process starts with an in-plane elastic shearing and is followed by plastic lattice relaxation (for more details see annual report of “Physical Characterization (XRD)”) [2,3]. Our results highlight a pathway for engineering and patterning of periodic ferroelectric domain structures.

Beyond engineering the domain pattern itself, epitaxial strain also allows tuning the phase transition temperatures of a ferroelectric material. This enables, e.g., a shift of the transition temperature close to room temperature and thus profit from the enhanced piezoelectric properties around the phase boundary. For fully strained $K_xNa_{1-x}NbO_3$ films on $TbScO_3$ with $0.60 < x < 0.80$, the transition temperatures were identified with XRD (collaboration with P.-E. Janolin, CNRS Centrale Supélec) and PFM revealing the possibility to induce shifts by more than 300 K with respect to the Curie temperature of a $K_xNa_{1-x}NbO_3$ bulk crystal.

To test the impact of strain and composition on the macroscopic piezoelectric coefficients d_{33} of $K_xNa_{1-x}NbO_3$, thin films were grown on $SrRuO_3$ bottom electrode layers, which were previously deposited on the substrates. Platinum top electrodes of different sizes were applied at the University Paris Sud (Dr. Sylvia Matzen). Using double beam laser interferometry, first macroscopic d_{33} values of (20 ± 5) pm/V could be evaluated by analyzing the small-signal amplitude for 20 to 60 nm thin films on $SrRuO_3/TbScO_3$. This value is quite comparable to that of lead-oxide based thin films.

However, the desired measurements of the large-signal amplitude could not be realized yet, since leakage currents in the films are still too high prohibiting the application of higher electric fields.

This was the starting point of the master thesis of Daniel Pfützenreuter that was finished in February 2018. In literature, the aliovalent doping of $K_xNa_{1-x}NbO_3$ thin films was described as an adequate way to reduce the leakage current. For this purpose, Cu- and Mn-doped films were deposited. Since PLD provides higher flexibility, $K_{0.5}Na_{0.5}NbO_3$ targets with different Cu and Mn doping concentration were used. At first, it was necessary to develop a suitable procedure for the preparation of dense, stoichiometric targets in cooperation with the group "Chemical and Thermodynamic Analysis" at the IKZ.

It appeared that the grinding of the raw powder mixture is an essential parameter in the target preparation. In contrast to the manual grinding of the ground powders by a mortar, use of a mixer mill leads to significantly smaller grains and a more homogeneous distribution (Fig. 2). We found that a mean grain size not exceeding 11 µm of the ground powder is necessary to achieve sufficiently dense and homogeneous targets. Differential thermal analysis showed that the addition of CuO and MnO_2 to the raw powder mixture, consisting of K_2CO_3, Na_2CO_3, and Nb_2O_5, has a significant impact on the solid-state reaction of the investigated material composition while sintering. Inductive coupled plasma optical emission spectroscopy (ICP-OES) measurements revealed that an alkaline excess in the raw powder mixture is necessary to obtain a stoichiometric target composition after sintering because potassium and sodium are very volatile at elevated temperatures. In conclusion, dense and phase pure Cu- and Mn-doped $K_{0.5}Na_{0.5}NbO_3$ targets can be achieved with an alkaline excess of 5 % and a two-stepped sinter routine where targets are annealed at 850° C for 6 h and at 1050 °C for 3 h by applying a heating rate of 5 K/min.

The results have shown that well-ordered ferroelectric, nearly stoichiometric Cu- and Mn-doped $K_{0.5}Na_{0.5}NbO_3$ thin films can be successfully grown on $DyScO_3$ substrates from such targets. The incorporated epitaxial lattice strain was verified by high-resolution XRD (HRXRD) measurements, and ferroelectric behavior was shown with PFM measurements. However, further electrical measurements are necessary to investigate the reasons for the leakage current and the possibilities for its reduction. We continue this study in cooperation with the group "Physical Characterization" in the framework of the projects "Bastet" (Leibniz Competition) and "Applikationslabor für Materialien der Oxidelektronik" (EFRE).

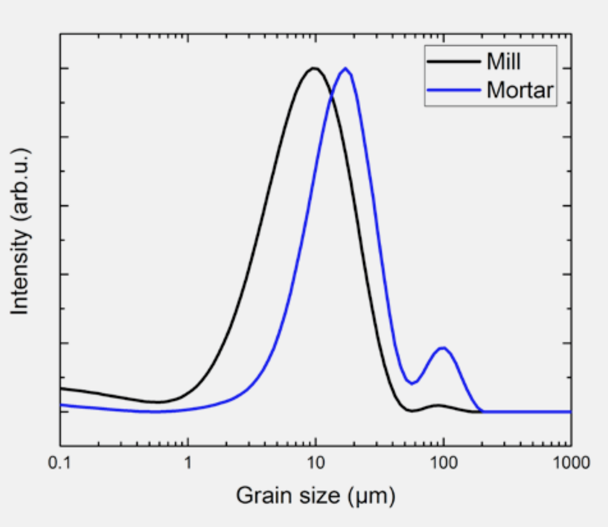

Fig. 2

Distribution of the grain size of ground powders after treatment with a mortar (blue curve) and a mixer mill (black curve). Particle sizes were measured by laser scattering.

Homoepitaxial $SrTiO_3$ thin films

In 2017, we have started in the framework of the Leibniz ScienceCampus GraFOx with the homoepitaxial growth of $SrTiO_3$ films with the aim to understand the role of defects in perovskite thin films and to control them. For this purpose, nearly perfect layers are required. Therefore about 10 - 15 nm thick $SrTiO_3$ films have been grown at first with PLD using a wide range of different growth parameters. Especially, we analyzed the influence of the substrate temperature (600 °C and 800 °C), the chamber pressure (10^{-1} - 10^{-5} mbar) and the laser energy (1 – 2.2 J/cm^2). To distinguish between effects due to the lack of oxygen and effects due to the scattering of the plasma particles with the background gas, we used either pure oxygen or argon atmosphere.

We found that the vertical lattice parameter of the $SrTiO_3$ thin films (which is an indicator for film off-stoichiometry) approaches the bulk value only for a high chamber pressure for both oxygen and argon process gas. To analyze film and interface quality in more detail, the group "Electron Microscopy" recorded scanning transmission electron microscopy (STEM) high and low angle annular dark field (ADF) images. Low angle ADF, sensitive to electrons diffusely scattered at point defects, reveals that this scattering is greatly enhanced for films grown at low pressure (< 10^{-2} mbar) (Fig. 3 (b)), while the film is almost indistinguishable from the substrate at 10^{-1} mbar (Fig. 3 (a)). As a first result, we conclude that high pressure during growth decreases the point defect density. However, in high angle ADF, which is sensitive to the atomic number and thus film stoichiometry, it is apparent that a high oxygen pressure leads to a non-stoichiometric interface as can be seen in Fig. 3 (b) as a pronounced black line at the interface, indicating missing strontium columns at the interface.

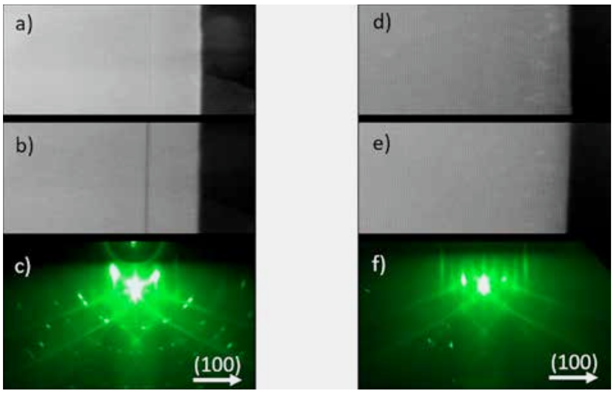

Fig. 3
Homoepitaxial $SrTiO_3$ thin films grown at 0.1 mbar oxygen pressure on two differently reconstructed surfaces. Low angle ADF images shown in (a) and (d) point in both cases to a very low point defect density. However, high angle ADF images show that the interface crucially depends on the substrate surface reconstruction, as the black line in (b) originates from a titanium-rich surface reconstruction, whose RHEED pattern is shown in (c). On a stoichiometric (2x1) reconstruction (f) the interface and the film is invisible also in high angle ADF (e).

Reflection high-energy electron diffraction (RHEED) investigations showed that the origin of the non-stoichiometric interface is a titanium-rich surface reconstruction of the $SrTiO_3$ substrate emerging at these conditions. It was found that this surface reconstruction (see Fig. 3 (c)) consists of a double layer TiO_2, which explains the visibility of the interface in high angle ADF. Under heating to temperatures > 800 °C in vacuum, the surface reorganizes and forms a (2x1) surface reconstruction (Fig. 3 (f)), which consists only of a TiO_2 monolayer and is stable under subsequent oxygen inlet. Homoepitaxial growth on a such prepared substrate at 600 °C and 800 °C substrate temperatures yields an almost indistinguishable film and interface as seen in Fig. 3 (d) and (e) for films growth at 800 °C.

In the future, these films will be compared to $SrTiO_3$ thin films grown epitaxially by MOVPE, to allow for detailed analysis of the corresponding incorporated defects.

$SrTiO_3$ thin films are also used in combination with $SrRuO_3$ thin films for the application as an ultrafast shutter in grazing incidence X-ray diffraction. These measurements have been performed within cooperation of Helmholtz-Zentrum Berlin (HZB), Universities of Potsdam and Hamburg [4].

Niobium oxide thin films

Within the project funded by the Leibniz Competition “Fundamentals of defects in oxides for resistive switching devices“, we have grown Nb_2O_5 and NbO_2 thin films using MOVPE and PLD, respectively. We aim to obtain single-crystalline niobium oxides to investigate point defects and their role in resistive switching behavior. Both Nb_2O_5 and NbO_2 exhibit many polymorphs. The most important ones for Nb_2O_5 are the T-Nb_2O_5 and H-Nb_2O_5 phases, which can be stabilized at low and high temperatures, respectively. Initial growth experiments were conducted with the MOVPE technique to establish a growth window for the different polymorphs. Figure 4 shows XRD scans of samples grown at different temperatures on (100) oriented $SrTiO_3$. For temperatures below 400 °C the layer remained amorphous, whereas the T-Nb_2O_5 was obtained for slightly higher temperature. Around 600 °C both the T-Nb_2O_5 and the H-Nb_2O_5 phase are obtained while a pure H-Nb_2O_5 phase is obtained for temperatures of 700 °C. Detailed analysis of the T-Nb_2O_5 films by atomic force microscopy (AFM) and HRXRD revealed that they contain grains with four different in-plane orientations that are related to the low symmetry of the T-Nb_2O_5 phase. The number of domain orientations could be reduced to two by employing (110) oriented $SrTiO_3$ substrates. However, the formation of domains in Nb_2O_5 thin film is intrinsically related to the low symmetry of the Nb_2O_5 phases and therefore challenging to overcome using epitaxy. To enable homoepitaxial films and therefore higher crystalline quality in future, the growth of Nb_2O_5 single-crystals is currently under investigation in the group “Oxides & Fluorides”.

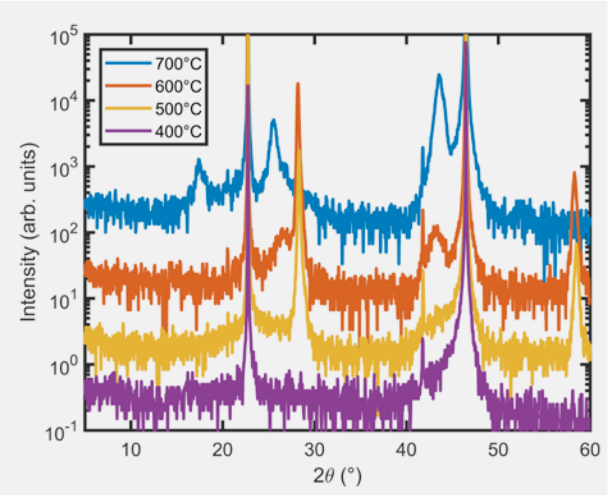

Fig. 4
HRXRD profiles of Nb_2O_5 films grown on (100) oriented $SrTO_3$ at different substrate temperatures.

The tetragonal phase of NbO_2, on the other hand, is very similar to the crystal structure of TiO_2. NbO_2 thin films have been grown on (110) oriented TiO_2 substrates using PLD. During the deposition, an Ar atmosphere was used to prevent the formation of Nb_2O_5. HRXRD investigations showed that the NbO_2 thin films had a single out-of-plane and a single in-plane orientation. Using these films, it was possible to determine the anisotropic optical properties of NbO_2 for the first time. Diffusion of Ti from the substrate to the film has prevented the investigation of the electrical properties yet. However, using buffer layers in the future should solve this problem.

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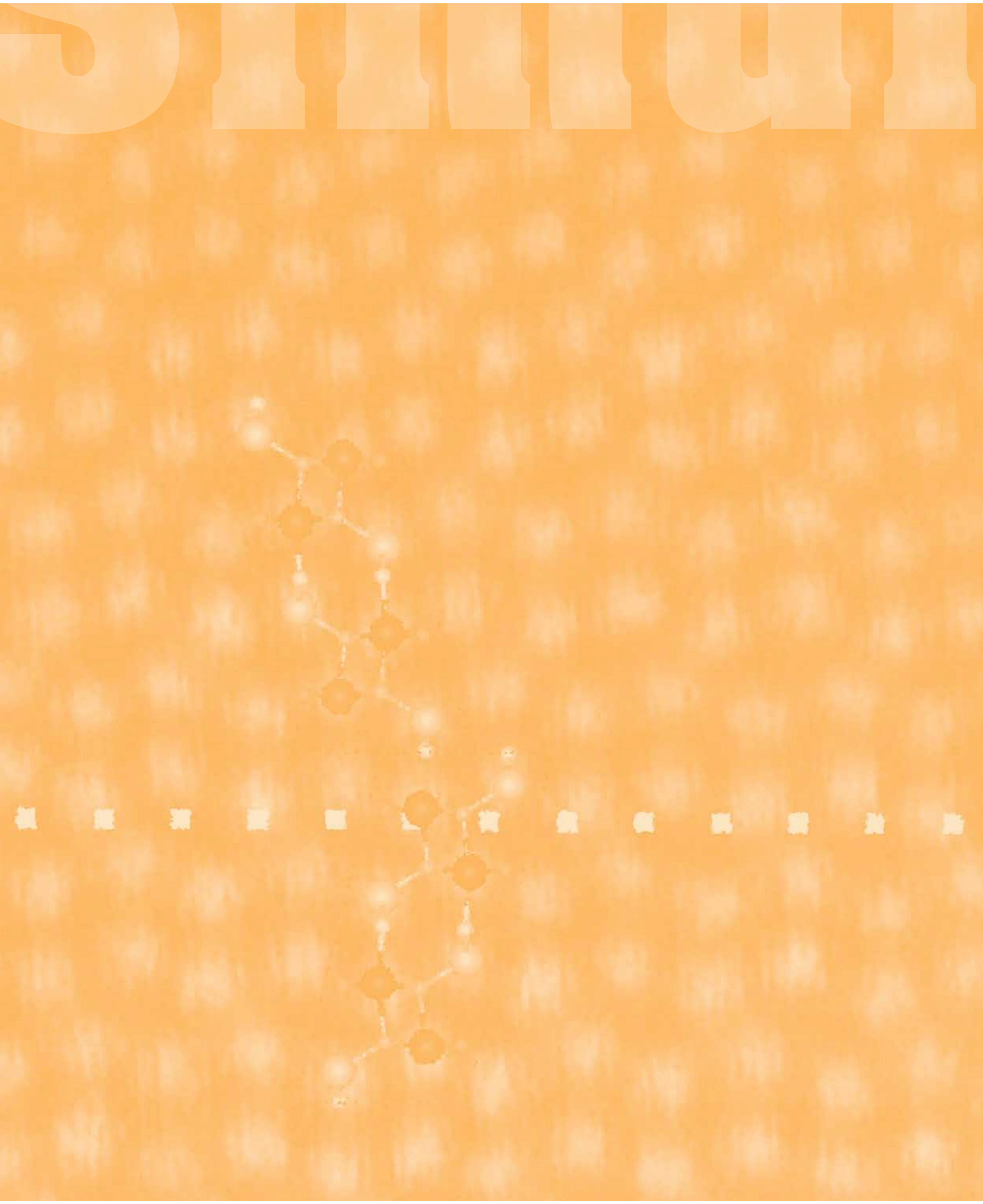
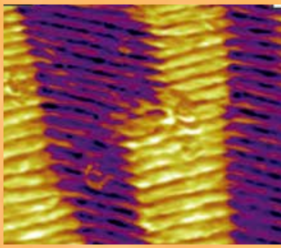
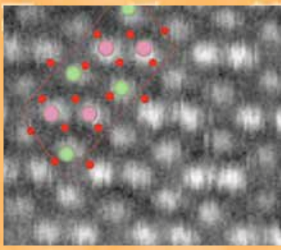
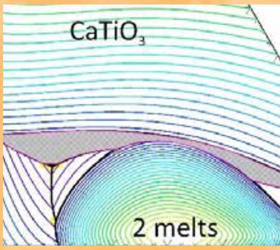
Simulation & Charakterisierung



Simulation & Characterization







Simulation & Charakterisierung

Head of department: Dr. Martin Albrecht

Der Schwerpunkt der Abteilung „Simulation und Charakterisierung“ liegt auf dem wissenschaftlichen Service für das Institut und externe Partner, der Grundlagen- und angewandten Materialforschung auf dem Gebiet der kristallinen Materialien, der Entwicklung und Anpassung wissenschaftlicher Methoden zur Charakterisierung von am Institut gewachsenen Materialien und der Entwicklung von Oberflächenpräparationstechniken.

Die Unterstützung der Kristallzüchtungsabteilungen und externer Partner durch wissenschaftlichen Service umfasst die routinemäßige Charakterisierung der elektrischen, optischen und strukturellen Eigenschaften, die Herstellung von Kristallkeimen und Substraten, die eine kristallografische Orientierung durch Röntgenbeugung, Schneiden und Oberflächenvorbereitung erfordert.

Die umfassenden Charakterisierungstechniken sowie das fundierte Wissen über atomare und erweiterte Defekte, die Analyse von Wachstums- und Entspannungsprozessen und Simulationsmethoden bieten einzigartige Möglichkeiten, Grundlagenforschung an den im Institut gewachsenen Materialien durchzuführen.

Die Gruppen betreiben auch eigene explorative Forschung in ihrem Fachgebiet mit nationalen und internationalen Partnern aus Wissenschaft und Industrie.

Zur Zeit bilden vier Arbeitsgruppen die Abteilung:
- *Physikalische Charakterisierung*
- *Elektronenmikroskopie*
- *Kristallbearbeitung*
- *Chemische und thermodynamische Analyse*

Simulation & Characterization

Emphasis of the division “Simulation and Characterization” is on scientific service for the institute and external partners, fundamental and applied materials research in the field of crystalline materials, development and adaption of scientific methods for characterization of materials grown at the institute and development of surface preparation techniques.

Support of the crystal growth departments and external partners by scientific service includes routine characterization of electrical, optical and structural properties, the preparation of seed crystals and substrates, which requires crystallographic orientation by X-ray diffraction, cutting and surface preparation.

The comprehensive characterization techniques, as well as the profound knowledge in atomic and extended defects, in the analysis of growth and relaxation processes and in simulation methods, offer unique opportunities to perform fundamental research on the materials grown in the institute.

The groups also perform own explorative research in their field of expertise with national and international partners both from academy and industry.

At the moment four research groups form the division:
- Physical Characterization
- Electron Microscopy
- Crystal Machining
- Chemical and Thermodynamical analysis

Simulation & Characterization:
Physical Characterization

Head Dr. Klaus Irmscher
Team K. Banse, A. Fiedler, I. Gamov, A. Kwasniewski, M. Pietsch, Dr. habil. M. Schmidbauer, J. Stöver

Überblick

Die Gruppe Physikalische Charakterisierung bietet wissenschaftlichen Service durch die Untersuchung wachstumsrelevanter physikalischer Eigenschaften der Kristalle mittels Röntgenbeugung, optischer Spektroskopie und Bildgebung, elektrischer Messungen und verwandter Techniken. Darüber hinaus sind wir für die Wartung und Modernisierung der Messgeräte sowie für die Entwicklung spezieller Messtechniken verantwortlich. In den Schwerpunktthemen des IKZ führen wir detaillierte Untersuchungen grundlegender sowie anwendungsrelevanter physikalischer Eigenschaften kristalliner Materialien durch. Insbesondere haben wir unsere Aktivitäten im Rahmen der mit dem „Applikationslabor für Materialien der Oxidelektronik“ und dem Leibniz ScienceCampus GraFOx (Growth and Fundamentals of Oxides for Electronic Applications) neu geschaffenen Bedingungen für die Grundlagen- und angewandte Forschung an kristallinen Oxidmaterialien verstärkt.

Die elektrische Charakterisierung (Hall-Effekt, C-V, DLTS, Raman-Spektroskopie) von β-Ga_2O_3*-Schichten, die in der Gruppe „Halbleitende Oxidschichten“ mittels metallorganischer Gasphasenepitaxie gezüchtet werden, war im Berichtszeitraum Teil einer laufenden Doktorarbeit und hat wesentlich zur Optimierung der Wachstumsbedingungen beigetragen [1]. Zwei weitere Doktoranden haben Mitte 2017 ihre Arbeit aufgenommen:*

- *Im Rahmen des Projekts „Physik und Kontrolle von Defekten in Oxidschichten für die adaptive Elektronik“, werden epitaktische* $SrTiO_3$*- und* Nb_2O_5*-Dünnschichten aus der Gruppe „Ferroelektrische Oxidschichten“ elektrisch und spektroskopisch mit dem Ziel charakterisiert, die für das resistive Schalten relevanten Punktdefekte zu identifizieren. Das Projekt wird im Rahmen des Leibniz-Wettbewerbs gefördert.*
- *Im Rahmen des DFG-Projekts „Elektromechanische Eigenschaften und atomarer Transport in AlN-Volumenkristallen bei hohen Temperaturen“ sollen Aluminiumnitridkristalle hinsichtlich ihrer piezoelektrischen Hochtemperatureigenschaften optimiert werden. Insbesondere zur Minimierung der elektrischen Verluste ist es erforderlich, Punktdefektarten und -konzentrationen zu identifizieren und kontrollieren. Das Projekt wird in Kooperation mit der Technischen Universität Clausthal durchgeführt.*

Zur Modernisierung unserer Messanlagen wurde 2017 ein UV/Vis/NIR-Spektrometer (Lambda 1050, PerkinElmer) aus Mitteln des EQuiLa-Projekts („Erforschung und Qualifizierung innovativer Lasermaterialien und -kristalle“) im Rahmen des Aufbaus des Zentrums für Lasermaterialien am IKZ erworben. Das Spektrometer bietet winkelabhängige Transmissions- und Reflexionsmessungen ohne Probenwechsel sowie eine hohe Empfindlichkeit. Es wird gemeinsam vom Zentrum für Lasermaterialien und unserer Gruppe genutzt. Weiterhin wurden im Rahmen des von EFRE geförderten Applikationslabors für Materialien der Oxidelektronik ein Deep-Level-Transient-Spektrometer (FT 1230, PhysTech) mit einem Kryostaten mit geschlossenem Kühlkreislauf (20 - 800 K) und ein 266 nm-cw-Laser (CryLaS) zur verbesserten (photo-)elektrischen Charakterisierung von halbleitenden Oxidkristallen erworben.

Ein weiterer Schwerpunkt unserer Gruppe ist die Charakterisierung ferroelektrischer Oxidschichten mittels Röntgenbeugung in Zusammenarbeit mit der Gruppe „Ferroelektrische Oxidschichten“. Diese Arbeit hat wesentlich zum Verständnis der Domänenstruktur in gitterverspannten Oxidschichten beigetragen. In diesem Zusammenhang wurden an modernen Synchrotronstrahlungsquellen (ESRF, BESSY II) anspruchsvolle Röntgenbeugungsexperimente durchgeführt. Unter den Doktoranden und Masterstudenten, die bei der Durchführung und Auswertung umfangreicher Röntgenbeugungsexperimente im Rahmen ihrer Qualifizierungsarbeit detailliert betreut werden, verteidigte 2017 Dorothee Braun erfolgreich ihre Doktorarbeit und Christoph Feldt erwarb seinen Master-Abschluss.

Da viele wichtige Ergebnisse in den Einzelberichten der jeweiligen Kristallwachstumsgruppen kommuniziert werden, konzentrieren wir uns hier auf zwei ausgewählte Themen unserer Arbeit:

- *Röntgenbeugungsuntersuchungen monokliner ferroelektrischer Domänen in* $K_{0.9}Na_{0.1}NbO_3$*-Dünnschichten und*
- *elektronische Raman-Streuung in n-Typ β-*Ga_2O_3*.*

Overview

The group “Physical Characterization” provides scientific service by investigating growth relevant physical properties of the crystals using X-ray diffraction, optical spectroscopy and imaging, electrical measurements, and related techniques. We are also responsible for maintenance and upgrading of measurement facilities as well as for developing dedicated measurement techniques. Moreover, we investigate fundamental and application-relevant physical properties of crystalline materials within the key research subjects of IKZ.

In particular, we have enforced our research activities within the recently established framework for applied and fundamental research on crystalline oxide materials, the Application Laboratory for Materials of Oxide Electronics (Applikationslabor für Materialien der Oxidelektronik) and the Leibniz ScienceCampus GraFOx (Growth and Fundamentals of Oxides for Electronic Applications).

In the reporting period, the electrical characterization (Hall effect, C-V, DLTS, Raman spectroscopy) of β-Ga_2O_3 layers, which are grown in the group "Semiconducting Oxide Layers" by means of metal-organic vapor phase epitaxy (MOVPE), has been part of an ongoing doctoral thesis and has contributed significantly to the optimization of the growth conditions [1]. Two new PhD students started their work mid 2017 on the following topics:

- In the project "Physics and control of defects in oxide films for adaptive electronics", funded in the frame of the Leibniz Competition, epitaxial $SrTiO_3$ and Nb_2O_5 thin films grown by the group "Ferroelectric Oxide Layers" are electrically and spectroscopically characterized with the goal to identify the point defects that have relevance for resistive switching.
- Within the DFG project "Electromechanical properties and atomic transport in AlN bulk crystals at high temperatures", aluminum nitride crystals shall be optimized with respect to their high-temperature piezoelectric properties. In particular, for minimizing electrical losses point defect species and concentrations have to be identified and controlled. This is a joint project together with the Technical University Clausthal.

Upgrading our measurement facilities, in 2017 a UV/Vis/NIR Spectrometer (Lambda 1050, PerkinElmer) was purchased in the framework of establishing the Center for Laser Materials at IKZ from the funds of BMBF "EQuiLa" project ("Erforschung und Qualifizierung innovativer Lasermaterialien und –kristalle", research and qualification of innovative laser materials and crystals). The spectrometer features angular dependent transmission and reflection measurements without sample change as well as high sensitivity. It is jointly used by the Center for Laser Materials and our group. Furthermore, a deep level transient spectrometer (FT 1230, PhysTech) with closed-cycle refrigerator cryostat (20 – 800 K) and a 266 nm-cw-laser (CryLaS) were acquired for improved (photo)electrical characterization of semiconducting oxide crystals in the framework of the EFRE project Application Laboratory for Materials of Oxide Electronics.

The other main focus of our group is the characterization of ferroelectric oxide layers by means of X-ray diffraction in cooperation with the group "Ferroelectric Oxide Layers". This work has contributed significantly to understanding the domain structure in strained oxide layers.

In this context, sophisticated X-ray diffraction experiments have been conducted at modern synchrotron radiation facilities (ESRF, BESSY II). Among the doctoral students and master students, who received careful supervision during execution and evaluation of extensive X-ray diffraction experiments as part of their qualification work, Dorothee Braun successfully defended her PhD thesis, and Christoph Feldt obtained his master degree in 2017.

Since many essential results are communicated in the individual reports of the respective crystal growth groups, we focus here on two selected topics of our work.

- X-ray diffraction from monoclinic ferroelectric domains in $K_{0.9}Na_{0.1}NbO_3$ thin films and
- electronic Raman scattering in n-type β-Ga_2O_3.

Results

X-ray diffraction from monoclinic ferroelectric domains in $K_{0.9}Na_{0.1}NbO_3$ thin films grown on (110) $NdScO_3$ substrates by MOCVD

We report on the ongoing investigation of $(K,Na)NbO_3$ epitaxial layers grown on (110) $NdScO_3$ (NSO) substrate (see Annual Report 2015). For a film thickness of 29 nm, a characteristic ferroelectric herringbone domain pattern is observed (Fig. 1 (a)) which is a consequence of coexisting a_1a_2 and M_C monoclinic phases. To correlate piezoelectric and structural properties, we performed grazing incidence in-plane X-ray diffraction (GIXD) at the European Synchrotron (ESRF). The corresponding two-dimensional X-ray diffraction pattern (Fig. 1 (b)) measured in the vicinity of the $(008)_{NSO}$ in-plane reciprocal lattice point exhibits:

- a peak splitting (P_1,P_2), which is associated with the a_1a_2-phase exhibiting in-plane monoclinic shearing with corresponding opposite shear angles ±β;
- prominent satellite peaks in the vicinity of P_1 and P_2, which are caused by the lateral periodicity of the domain pattern and the alignment of the domain walls leading to a corresponding orientation of the satellite branches (marked as blue dashed lines).

We have confirmed this tentative interpretation by simulations based on kinematical scattering theory. The scattered intensity from a periodic array of domains can be expressed as: [2]

$$I(\boldsymbol{Q}) = \left|\sum_{k=1}^{N} F_k(\boldsymbol{Q})\, exp(-i\boldsymbol{Q}\boldsymbol{r}_k)\right|^2 \cdot G(\boldsymbol{Q}) \qquad (1)$$

The first factor of the product describes coherent scattering from a single pair of 'violet' and 'orange' domains (Fig. 1 (c)) consisting of N pseudocubic (pc) unit cells (at positions r_k) and corresponding structure amplitudes $F_k(\boldsymbol{Q})$. The second factor of the product takes into account the periodicity of the domain pattern. Based on Eq. (1) an exemplary simulation around the $(008)_{NSO}$ reciprocal lattice point is shown in Fig. 1 (d) for $(100)_{pc}$ oriented domains with sizes of 29 nm and 100 nm along the $[001]_{NSO}$ and $[110]_{NSO}$ directions, respectively.

For a single pair of 'orange' and 'violet' domains we have chosen in-plane domain wall angles of $\alpha = \pm 15°$ and corresponding monoclinic distortions of the pseudocubic unit cell of $\beta = \pm 0.12°$ (see Fig. 1 (c)). For a mean domain periodicity of L = 30 nm with a standard deviation of σ = 5 nm, we achieve good agreement between experiment and simulation.

The elastic strain energy density is degenerated for the two $(100)_{pc}$ and $(001)_{pc}$ pseudocubic orientations. Nevertheless, a distinctive hierarchy of domain evolution is observed with exclusive in-plane a_1a_2 domains for very thin films and the retarded onset of a ferroelectric M_C phase at larger film thickness. With increasing thickness the domain pattern transforms from stripe domains to a herringbone pattern and, eventually, to a checkerboard-like structure. During that transformation, the fraction of the M_C domains is steadily increasing. The observed behavior is caused by the energy balance of depolarization field and anisotropic strain relaxation in the film.

This is exemplarily depicted in Fig. 2 (a) and (b) where the domain structure is presented for two samples with thicknesses of 38 nm and 52 nm, respectively. Along with morphological changes of the domain pattern we observe an additional peak (P_3) in the GIXD diffraction patterns (Fig. 2 (c), (d)) which is caused by the M_C domains. For the M_C phase, plastic strain relaxation is throughout observed. By contrast, the a_1a_2 domains show a two-step strain relaxation mechanism starting with an in-plane elastic shearing. At 52 nm thickness, we observe the onset of plastic lattice relaxation.

Our results highlight a pathway for engineering and patterning of periodic ferroelectric domain structures. They have been obtained in close collaboration with the group "Ferroelectric Oxide Layers" and have been recently published. [2, 3]

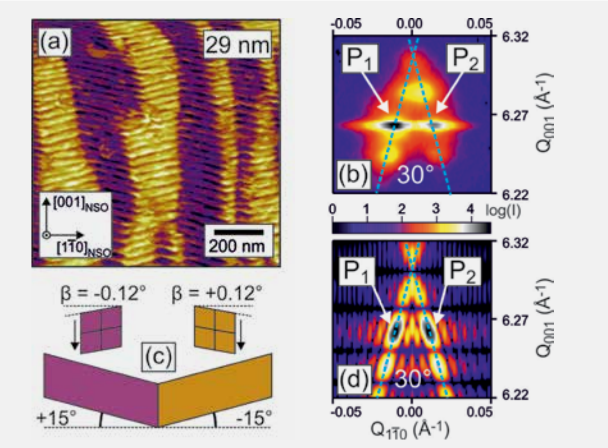


Fig. 1

(a) Ferroelectric domain pattern of a 29 nm $K_{0.9}Na_{0.1}NbO_3$ film grown on (110) $NdScO_3$ substrate. (b) Experimental GIXD in-plane intensity distribution in the vicinity of the $(008)_{NSO}$ substrate Bragg reflection. (c) Structural model and (d) Corresponding simulation.

Electronic Raman scattering in n-type β-Ga_2O_3

Monoclinic gallium sesquioxide (β-Ga_2O_3) belongs to the transparent semiconducting oxides. It is distinguished by its large band gap of about 4.7 eV, which is the reason for a transparency range extending deep into the ultraviolet and for a high electrical break down field estimated at 8 MV/cm. Combined with the feasibility of n-type doping by Si, Sn or Ge, β-Ga_2O_3 has great potential as a material for solar-blind photo detection and power electronics. For these applications, doping control is of outermost importance. Recently, however, it was questioned whether the mentioned donor species really act as simple, effective mass donors or form so-called DX centers, which would be associated with self-compensation and would significantly reduce the doping efficiency.

To elucidate the fundamental donor doping behavior, we investigated bulk crystals grown by the Czochralski method [4] in the group "Oxides & Fluorides" doped during the growth with Si and Sn, respectively. Besides the electrical characterization by Hall effect measurements, we performed Raman spectroscopy.

The depolarized Raman spectra of Si doped β-Ga_2O_3 crystals, including a semi-insulating one, in Fig. 3 are dominated by the familiar lines due to scattering at the Raman active phonon modes. For degenerately doped crystals (Mott criterion: $n > 3 \times 10^{18}$ cm^{-3}), we observe additional peaks at 256 cm^{-1} and 282 cm^{-1}, forming a double peak, and at about twice the wavenumber of the second peak (563 cm^{-1}). Since these three new signals are apparently independent of the excitation wavelength (see Fig. 4), we can exclude that they originate from photoluminescence. In fact, they must be due to inelastic scattering of the exciting photons.

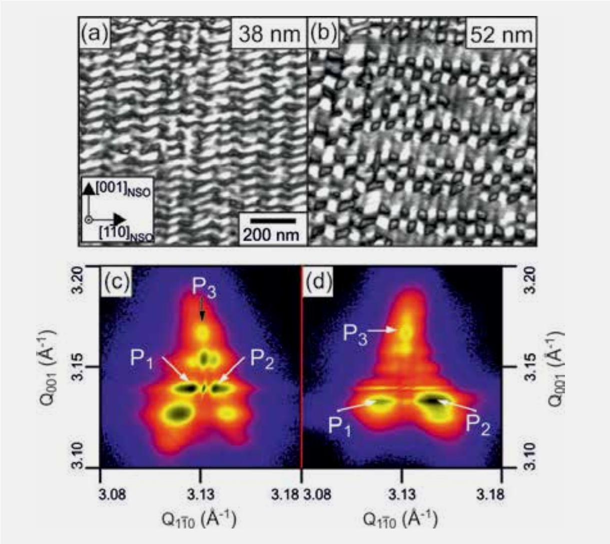


Fig. 2

Ferroelectric domain patterns of (a) 38 nm and (b) 52 nm $K_{0.9}Na_{0.1}NbO_3$ thin films along with corresponding experimental GIXD in-plane intensity distributions in the vicinity of the $(2\text{-}24)_{NSO}$ substrate Bragg reflection ((c), (d)).

Furthermore, the additional lines appear in the spectrum independent of the shallow donor doping species, Sn or Si, essentially at the same positions, as can be seen in Fig. 5. This means, that they cannot be due to scattering at local vibrational modes involving either Sn or Si because of the huge mass difference between both atoms, which would lead to a large difference in the vibrational frequency. Therefore, we attribute the doping induced Raman features to electronic Raman scattering. We assume that the scattering process is accompanied by the excitation of electrons from an effective-mass like donor impurity band into the conduction band. This assignment is based on the facts that the high-energy edge of the double peak coincides with the ionization energy expected for effective-mass like donors in β-Ga_2O_3 (≈ 36 meV, cf. [5]), and that the Raman signals only appear for doping concentrations exceeding the Mott criterion, i.e. ,after the formation of an impurity band. The latter leads to metallic-like conduction with a nearly temperature independent Fermi level located inside the impurity band and explains why we observe only a weak temperature dependence of the line shape and position. The high-frequency Raman line at $563\ \mathrm{cm}^{-1}$ (≈ $2 \times 282\ \mathrm{cm}^{-1}$) may be explained by second-order electronic Raman scattering, i.e., an incident photon excites in two subsequent scattering events two electrons.

In conclusion, our Raman spectroscopic investigations provide strong evidence that both Si and Sn behave as effective mass-like donors in β-Ga_2O_3 although they preferentially replace Ga in tetrahedral and octahedral coordination, respectively.

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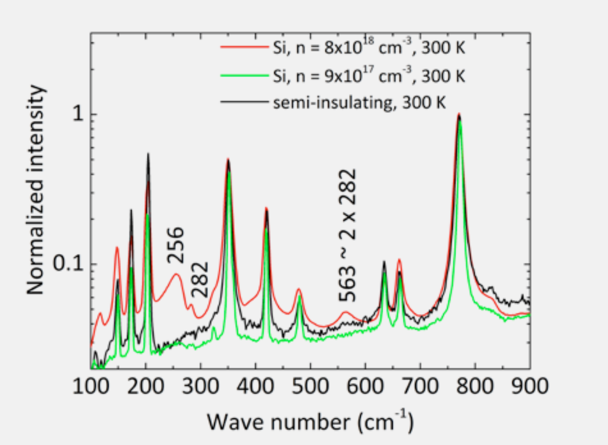

Fig. 3

Raman spectra of n-type (Si doped) and semi-insulating (Mg doped) β-Ga_2O_3 single crystals at room temperature. In addition to the familiar lines due to scattering at the Raman active phonon modes of β-Ga_2O_3, three peaks (labeled by their wavenumbers) appear in the spectrum of the degenerately doped sample (n = $8 \times 10^{18}\ \mathrm{cm}^{-3}$, red curve).

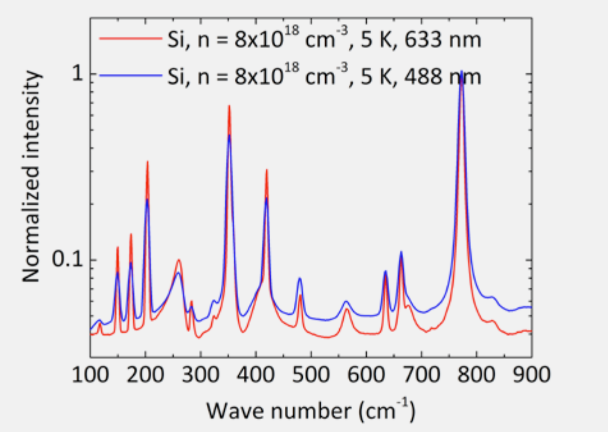

Fig. 4

Raman spectra of a heavily n-type doped β-Ga_2O_3 sample for two different excitation wavelengths (633 nm and 488 nm). The three additional lines (cf. Fig. 3) preserve their positions at 256, 282, and 563 cm^{-1} and, hence, must be due to an inelastic scattering process.

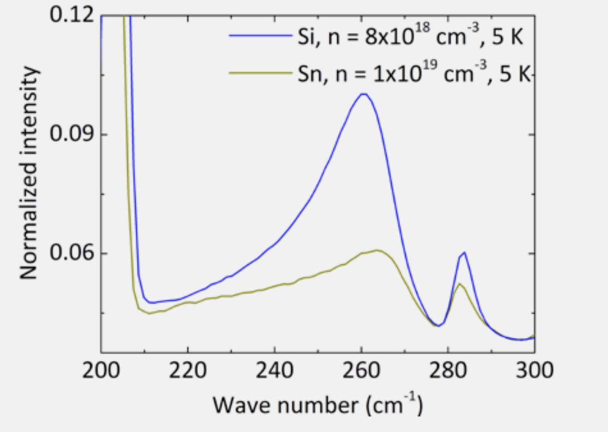

Fig. 5

Raman spectra of β-Ga_2O_3 crystals which were heavily doped with Si and Sn, respectively. The three additional Raman lines are essentially independent of the kind of donor impurity (only the two low-frequency lines are shown here on an enlarged scale).

Simulation & Characterization:
Electron Microscopy

Head Dr. Martin Albrecht
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Überblick

Die Arbeitsgruppe Elektronenmikroskopie charakterisiert kristalline Materialien mit elektronenmikroskopischen Methoden sowohl im Rahmen des wissenschaftlichen Service als auch im Bereich der Grundlagenforschung. Thematischer Schwerpunkt ist der Zusammenhang zwischen physikalischen Eigenschaften und Struktur von Halbleitern und Oxiden. Die Methoden reichen von der Rasterelektronenmikroskopie (energie- und wellenlängendispersive Röntgenspektroskopie, Elektronenrückstreubeugung (EBSD), Kathodolumineszenz) über die Ionenstrahlbearbeitung bis zur Transmissionselektronenmikroskopie (aberrationskorrigierte Transmissionselektronenmikroskopie und Rastertransmissionselektronenmikroskopie mit atomarer Auflösung). Die Gruppe arbeitet eng mit Gruppen des Kristallwachstums, der ab-initio Modellierung und der Simulation zusammen. Neben der Standardcharakterisierung von Oberflächen und Zusammensetzung, der Phasenanalyse und der Analyse von Einschlüssen werden insbesondere grundlegende Arbeiten zu Wachstums- und Relaxationsprozessen epitaktischer Schichten sowie zu Kristalldefekten durchgeführt. Um bildgebende Verfahren zu verbessern und sie auf die spezifischen Probleme und die laufenden Arbeiten am Institut anzupassen, werden eigenständige methodische Arbeiten durchgeführt. Die Gruppe arbeitet in nationalen und internationalen Forschungsprojekten und Forschungsverbünden und ist maßgeblich am Leibniz-WissenschaftsCampus GraFOx beteiligt.

2017 wurden unsere Forschungsaktivitäten durch 5 Doktorandinnen und Doktoranden unterstützt. Das Projekt SPRInG, das von der Europäischen Union im Rahmen des Marie Curie-Sklodowska Programms „European Integrated Doctorate“ gefördert wird, hat zum Ziel Studierende zu gleichen Teilen in Industrie und akademischer Forschung auszubilden. Im Rahmen des französischen Exzellenz-Netzwerks GaNeX, hat Natalia Stolyarchuk ihre Promotion in einem gemeinsamen Verfahren der Humboldt-Universität zu Berlin und der Universität Côte d’Azur erfolgreich verteidigt.

Da einige der Ergebnisse der Elektronenmikroskopie in den Berichten der Arbeitsgruppen der Kristallzüchtung dargestellt werden, beschreiben wir im Folgenden einige ausgewählte Themen unserer derzeitigen Arbeit, die zusammen mit internen und/oder externen Partnern im Rahmen von gemeinsamen Forschungsprojekten bearbeitet werden. Es handelt sich dabei um Arbeiten:

- *zur Oberflächendiffusion beim homoepitaktischen Wachstum von β-Ga_2O_3(100), Arbeiten die im Rahmen einer Masterarbeit gemeinsam mit den Arbeitsgruppen „Halbleitende Oxidschichten“ und „Oxide & Fluoride“ durchgeführt wurden,*
- *zu den Rekombinationsprozessen in InGaN, die zusammen mit dem Max-Planck-Institut für Eisenforschung in Düsseldorf, der Universität Peking, dem Institut für Hochdruckphysik der polnischen Akademie der Wissenschaften, sowie dem Paul-Drude-Institut in Berlin und dem Max-Born-Institut in Berlin im Rahmen des EU-Projekts SPRInG durchgeführt wurden und*
- *zum Einfluss der Koordinationsumgebung von In und Ga auf die Phasenbildung im Sytem $(In,Ga)_2O_3$, die im Rahmen des Leibniz-WissenschaftsCampus GraFOx gemeinsam mit dem Fritz-Haber-Institut, dem Paul-Drude Institut und der Universität Leipzig durchgeführt wurden.*

Overview

The electron microscopy group performs scientific service and basic research in the field of characterization of crystalline material by means of electron microscopy. Primary focus is the relation between physical properties and structure of semiconductors and oxides. The methods cover the wide range from scanning electron microscopy (SEM), i.e., energy and wavelength dispersive spectroscopy, electron backscatter diffraction (EBSD), cathodoluminescence (CL) to transmission electron microscopy (TEM), i.e., aberration-corrected transmission electron microscopy and scanning transmission electron microscopy (STEM) with atomic resolution.

The team works in close collaboration with groups performing crystal growth, as well as ab-initio modeling and simulation. We perform standard characterization of surfaces and composition, phase analysis and analysis of inclusions as well as fundamental studies of growth and relaxation mechanisms and of defects. Besides this, methodological work is done to improve electron optical imaging techniques and adapt them to the specific problems at the institute. The group works in collaborative national and international research projects and is significantly involved in the Leibniz ScienceCampus GraFOx.

In 2017, our research activities were supported by five doctoral students. The SPRInG project, funded by the European Union under the Marie Curie-Sklodowska programme "European Integrated Doctorate", aims to train students equally in industry and academic research. Within the framework of the French Excellence Network GaNeX, Natalia Stolyarchuk successfully defended her doctorate in a joint procedure of the Humboldt University of Berlin and the University of Côte d'Azur.

As some of the results of the electron microscopy group are included in the reports of the groups working in crystal growth, in the following we will present some selected topics of our current work, which was performed together with the groups "Semiconducting Oxide Layers", "Oxides & Fluorides" in the institute and external partners in joint research projects. These are the following works:

- surface diffusion in the homoepitaxial growth of β-Ga_2O_3 – work carried out as part of a master's thesis in collaboration with the working groups "Semiconducting Oxide Layers" and "Oxides & Fluorides",
- recombination processes in InGaN performed together with the "Max-Planck-Institut für Eisenforschung" in Dusseldorf, the Institute for High Pressure Physics of the Polish Academy of Sciences, the Paul-Drude-Institut für Fetskörperelektronik in Berlin and the Max Born Institute for Nonlinear Optics and Short Pulse Spectroscopy in Berlin within the framework of the EU project SPRInG ("Short Period Superlattices for Rational (In,Ga)N"),
- the influence of coordination environment of In and Ga on the phase formation in the system $(In,Ga)_2O_3$ – work carried out within the scope of the ScienceCampus GraFOx together with the Fritz-Haber-Institut der Max-Planck-Gesellschaft, the Paul-Drude-Institut für Festkörperelektronik and the University of Leipzig.

Results

Surface diffusion in the homoepitaxial growth of β-Ga_2O_3 (100)

β-Ga_2O_3 has recently gained renewed scientific interest as a wide bandgap oxide semiconductor for future power electronic and optoelectronic applications. The β-polymorph of Ga_2O_3 is the thermodynamically most stable structure. It is monoclinic and has two distinct cleavage planes, namely (100) and (001). With a wide bandgap of Eg = 4.7 eV it is transparent up to the deep ultraviolet in the optical spectrum. In contrast to other wide band-gap semiconductors in the field (e.g., SiC and GaN), bulk single crystals of β-Ga_2O_3 can be grown with high structural perfection from the melt at relatively low production costs.

While recently progress has been achieved in homoepitaxial growth and the design of devices like Schottky barrier diodes and field-effect transistor (FET) structures, fundamental understanding of the homoepitaxial growth in this material system is still weak.

In our previous work [1], we showed that homoepitaxial growth by metal-organic vapor phase epitaxy (MOVPE) on (100) plane of β-Ga_2O_3 is challenging since twin lamellae form if an unproper miscut is chosen, and influence the electrical properties of the layer in a negative way. [2] According to our work [1], these twin lamellae form by a double positioning process through two-dimensional island nucleation, i.e., a layer-by-layer growth mode instead of the desired step-flow growth mode.

Due to a low surface diffusivity of the ad-atoms during growth, an unusual miscut angle as high as 6° is required to obtain defect-free materials. Diffusion behavior for ad-atoms has been intensively studied for metal-on-metal epitaxy [3], but also for semiconductors. For example, studies of the epitaxy for GaN revealed surface migration barriers of 2.48 eV while in the case of GaAs surface migration barriers are in the range of 1.1 - 1.3 eV.

One approach to determine the surface migration barriers for diffusion of ad-atoms experimentally is to investigate the temporal development of the size distribution and the density of two-dimensional islands during sub-monolayer growth and compare them with predictions by nucleation theory. Ideally, this requires a flat surface without surface steps. In case of conventional semiconductors, such conditions are hardly achievable since all real surfaces have a certain miscut, i.e., a finite step width. β-Ga_2O_3, with its monoclinic structure and two easy cleavage planes, offers the advantage to realize such surfaces by simple cleaving. Moreover, these cleaved surfaces are almost ideal since they are not subjected to any further surface preparation process.

Figure a) - e) show AFM images of the layer deposited on freshly cleaved surfaces by metal-organic vapor phase epitaxy after 15 s. Growth temperatures ranging from 750° C to 850 °C have been chosen. After growth, two-dimensional islands are present on the surface with a height of about 5.9 Å, corresponding to a half height of a β-Ga_2O_3 unit cell. The density of nucleated islands decreases with increasing temperature. To determine the diffusion length of ad-atoms, we measured the areal density of nucleated islands during the initial stages for four different temperatures. It is crucial to adjust the time frame in such a way that at the highest temperature islands are still present, while at the lowest temperature – where the highest density is expected – coalescence is avoided. Fig. 2 shows the experimentally determined island densities plotted against the reciprocal growth temperature.

Frankel and Venables proposed a phenomenological model to describe nucleation processes on surfaces by rate equations approach. [4] These authors simplify the computational effort by considering the mean ad-atom densities n_1 and the mean number of atomic clusters formed of I atoms N_i, instead of describing the local distribution of individual ad-atoms and islands. Considering a perfect surface, we derive the stable cluster density N in dependence of the incoming flux of atoms and the diffusion barrier E_D by the so-called Walton relation:

$$\frac{N}{N_0} = \eta(Z,i)Q(i)\left(\frac{R}{N_0 \alpha \mathrm{v}}\right)^p \exp\left\{\frac{E}{\mathrm{k_b T}}\right\}$$

By fitting the temperature dependent cluster-density by an Arrhenius plot, we obtain the activation energy E_a and the critical cluster size i. Since the activation energy is the sum of the formation energy of a stable cluster with *i* atoms and the activation energy for surface diffusion on the surface, we need to estimate the activation energy to form a stable cluster. The latter is obtained by using known parameters like the number of nearest neighbor "bonds" of Ga, their bonding energy E_b, and the heat of fusion of β-Ga_2O_3. From this, we estimate a cluster binding energy of E_i = 206 meV per Ga atom. Taking this cluster binding energy, we obtain a diffusion barrier for the diffusion of a Ga atom on the (100) surface of β-Ga_2O_3 of E_d = 1.54 eV. This activation energy for surface diffusion lies in between measured values for GaAs and GaN found in the literature. The diffusion coefficient at 850 °C, which we obtained from this activation energy, agrees perfectly with experimentally determined values from our previous work. [1] It will allow us to choose the right miscut for the desired growth temperature and thus to optimize the structural and electrical properties of our layers.

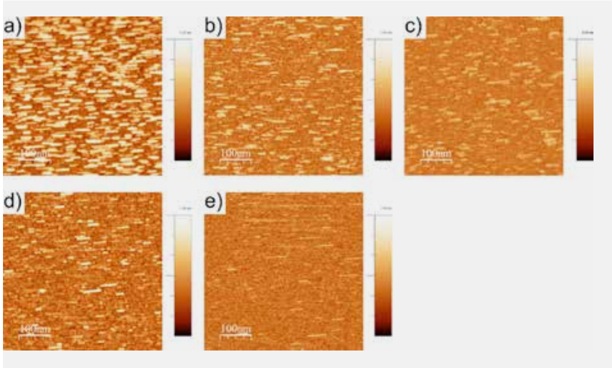


Fig. 1
Atomic force microscopic (AFM) image of a cleaved surface of (100) oriented β-Ga_2O_3 substrate after deposition for 15 s at 750 °C, 775 °C, 800 °C, 825 °C and 800 °C, respectively.

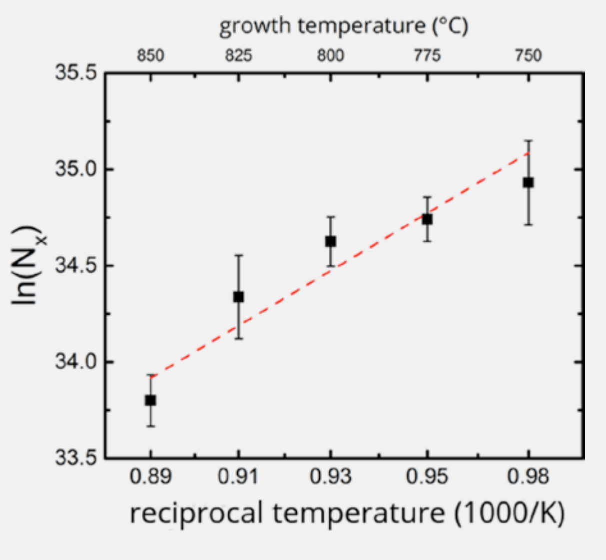


Fig. 2
Island density vs. reciprocal temperature. From the Arrhenius plot (indicated by the dashed red line) the activation energy is obtained. The intersection with the y-axis gives the stable cluster.

Recombination processes in InGaN studied in short period superlattices

Recent progress in growth and fabrication technology has enabled the demonstration of InGaN-based light-emitting diodes (LEDs) with maximum external quantum efficiencies of over 80% in the violet-blue spectral range. However, the performance of InGaN-based LEDs drops substantially for increasing emission wavelengths. For LEDs operating in the green and yellow spectral range, the maximum external quantum efficiency is remarkably lower: around 40–50% and 20%, respectively. In literature, this problem is known as the "green gap", which is present in both LEDs and laser diodes, pointing to some internal processes in the InGaN active region [5, 6, 7].

Overcoming such limitations requires a detailed understanding of radiative and non-radiative processes in the (In,Ga)N quantum wells – the key element for converting current into light. Within the framework of the EU project "SPRInG" *(Short Period Superlattices for Rational (In,Ga)N)* and in close collaboration with the Max Born Institute for Nonlinear Optics and Short Pulse Spectroscopy (Berlin) and the Max-Planck-Institut für Eisenforschung (Düsseldorf), we have examined the charge carrier recombination processes by means of time-resolved photoluminescence (PL) studies and density functional theory calculations. Our samples consist of polar (In,Ga)N quantum wells, stacked in a superlattice, as displayed in scanning transmission electron microscopy high angle annular dark field images in Figure 3. The composition and thickness of the quantum wells have been quantified by transmission electron microscopy studies, revealing a thickness of a single monolayer and an In content of 25 %.

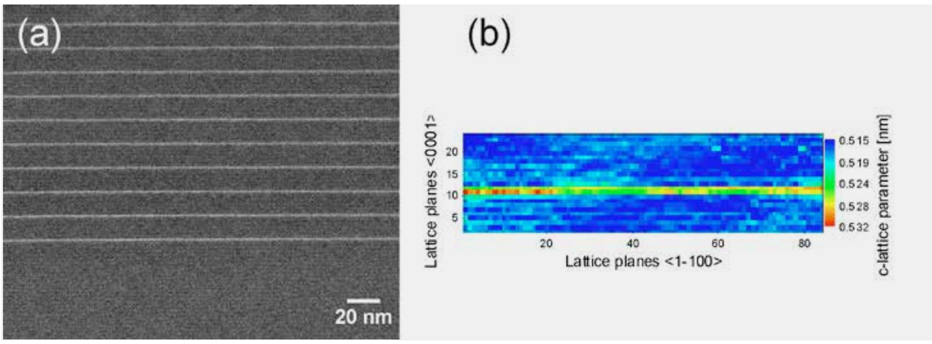
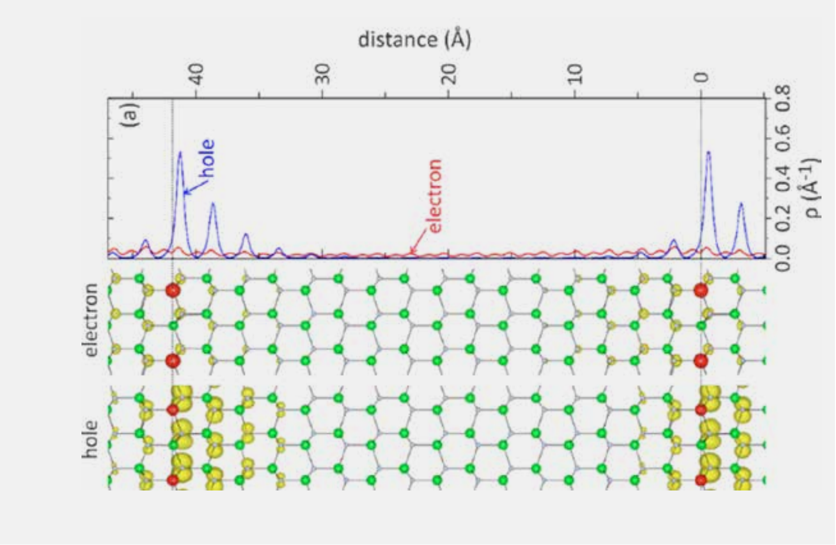


Fig. 3
(a) STEM-HAADF image of a single monolayer (In,Ga)N quantum well. (b) c-lattice parameter map of the monolayer region. Quantification yields an average In content of 25 %. (In,.Ga)N monolayers appear bright.

Since the thickness of our (In,Ga)N quantum wells of only one monolayer is substantially lower than conventional ones (typically 10 monolayers), two influencing factors affecting the emission of conventional quantum wells can be excluded: First, polarization fields causing a spatial separation of the charge carriers and second, electron localization due to the confinement of the quantum well. Hence, the complexity of our system is much lower compared to conventional ones allowing a fundamental examination of recombination processes in a model system.

Despite the absence of these two influencing factors we observe very similar recombination properties, as compared to conventional quantum wells, i.e., a so-called S-shape of the temperature dependent luminescence and a spectral dependence of the PL decay times.

Our observations, therefore, challenge the common knowledge that the S-shape and the spectral dependence of the carrier lifetime are essentially influenced by localization of electrons and holes at separate sites of the quantum well. Instead, we identified the localization of holes as a decisive factor governing charge carrier recombination. Reducing the hole localization, without changing the composition or thickness of the (In,Ga)N monolayer, can be achieved by decreasing the GaN barrier width from 50 to 6 monolayers (Fig. 4) Our experimental results show that reducing the hole localization directly enhances the influence of non-radiative transitions, reducing the efficiency of the light conversion (fig. 5). Our results thus point to increasing radiative recombination rates with increasing degree of hole localization.



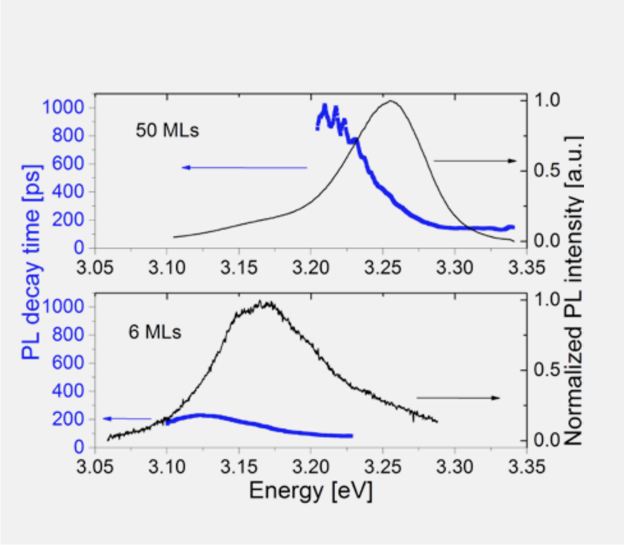


Fig. 4
Density functional theory calculations showing the charge densities of electrons (red) and holes (blue) of a single (In,Ga)N monolayer with an Indium content of 25 %.

Fig. 5
Spectral dependence of the decay time for the case of 50 ML and 6 ML GaN barrier.

The coordination environment and the phase stability of $(In_xGa_{1-x})_2O_3$

The interest in the group III sesquioxides (In_2O_3, Ga_2O_3, Al_2O_3) has grown significantly over the past few years because of their outstanding material properties. Due to their large band-gap combined with high breakdown field and efficient n-doping possibilities, they are classified as transparent semiconducting oxides and suited for a wide field of applications including high-power electronics and energy applications. To fully exploit their potential, alloying of the binaries is desired to tune their properties - especially the band-gap - over a wide range. Alloying of these oxide systems is however not straightforward since the binaries have different thermodynamically stable crystal structures [8], and for each of them a variety of different polymorphs have been reported. Therefore, it is interesting to investigate phase formation as a function of composition, to see the influence of growth conditions and to get an insight into the mechanisms that govern the phase stability. This was done in the framework of GraFOx joining activities of Leibniz-Institute für Kristallzüchtung, Paul-Drude-Institut für Festkörperelektronik, Universität Leipzig and Fritz-Haber-Institut der Max-Planck-Gesellschaft.

In the case of $(In_xGa_{1-x})_2O_3$,, the stable structures of the binary materials are monoclinic (C2/m) - also indicated as β-phase - for Ga_2O_3 and cubic bixbyite (Ia-3) for In_2O_3. Recent calculations of the phase diagram based on the cluster expansion methods show rather narrow stability windows with low solubility limits for the phases mentioned above at the Ga-rich and In-rich ends. Additionally, close to indium concentrations of x=0.5 the hexagonal $InGaO_3$(II) alloy phase [9] is found to be stable. It is important to note the different coordination environments of the cations in the different structures. In the monoclinic /hexagonal lattices, there is an equal amount of four/five-fold and six-fold coordinated positions, while in the cubic bixbyite structure all cations share the same octahedral (six-fold) coordination environment.

To compare these calculations with experimental findings, thin $(In_xGa_{1-x})_2O_3$ layers grown by pulsed laser deposition (PLD) on sapphire are studied by transmission electron microscopy (TEM). By using a simultaneously rotating target and substrate, layers with a continuously increasing indium content are deposited with indium content varying typically between $0<x<0.9$. For a sample grown at $T_g=680$ °C and $p(O_2)=3\cdot10^{-4}$ mbar, the spatially resolved crystallographic properties are retrieved by X-ray diffraction (XRD) ω-2θ scan and the results are displayed in Fig. 6 (a). This measurement shows that the monoclinic phase is stable up to $x<0.5$ with an increasing lattice parameter as the amount of incorporated indium increases. For higher indium contents, signs of both the β-, h- and c-phase are present in the XRD map, with the hexagonal phase dominating for $0.5<x<0.75$ and the cubic phase dominating at the indium-rich end. TEM measurement of a piece of the layer at the position indicated by the red arrow - which corresponds to overall indium content of x=0.75 - shows indeed that these three phases are present in the layer, in a layered fashion as shown in the TEM bright field image in Fig. 6 (b). The monoclinic and hexagonal $(In_xGa_{1-x})_2O_3$ phases consist of nanometer-sized grains while the cubic phase grows in larger domains of a few ten to hundred nanometers in size.

The indium content in the respective phases is determined by a STEM-EDXS measurement, using the Ga Kα and In Lα peak intensities. The results show that the indium content in the monoclinic phase can't exceed 50%, the hexagonal $(In_xGa_{1-x})_2O_3$ phase can be stabilized for In contents in the range x=0.50-0.70 and the cubic In_2O_3 lattice allows incorporation of gallium atoms up to approximately 10%.

High-resolution electron microscopy allows us to resolve the atomic pattern and determine the coordination environment of the gallium and indium atoms in the three phases observed for the alloy. Figure 7 shows STEM images of β-, h-, and c-$(In_xGa_{1-x})_2O_3$ alloys with indium concentrations of approximately x = 0.40, 0.60 and 0.90, respectively. In this technique, a scattering of the beam electrons by the atomic columns produces intensity resulting in their appearance as bright dots on a darker background in the images. Only the metal columns are visible since the light oxygen columns produce too little contrast to be observed. Additionally, since the scattering power scales roughly as Z^2, where Z is the atomic number, the contrast is sensitive to the average atomic number along the column, and thus, the presence of heavier indium atoms in a column will produce a higher intensity. In the β and h structures, a clear intensity difference in the differently coordinated columns is observed, indicating that the indium atoms are found to be preferentially incorporated in the 6-fold (octahedral, pink) lattice positions. The gallium atoms, which are more flexible in their coordination environment, are therefore mostly assigned to the 4-fold and 5-fold positions in the β- and h-structure respectively. In the cubic bixbyite $(In_xGa_{1-x})_2O_3$ lattice, all metal lattice sites have the same octahedral environment and there are only slight intensity variations due to the statistical incorporation of the gallium atoms.

The experimental observation of the preferential incorporation of indium atoms to octahedral lattice sites agrees with calculations by Maccioni et al.,[10] who have shown that an energy penalty of 1.1 eV exists for indium to go on tetrahedral lattice sites. In the case of the monoclinic phase, this explains why its formation is hindered for x>0.5: at this point, all octahedral sites are filled by indium atoms. In the hexagonal lattice, the strain is minimized when all tetrahedral/octahedral sites are occupied by gallium/indium atoms, which explains why this alloy structure is mostly stabilized at intermediate indium contents.

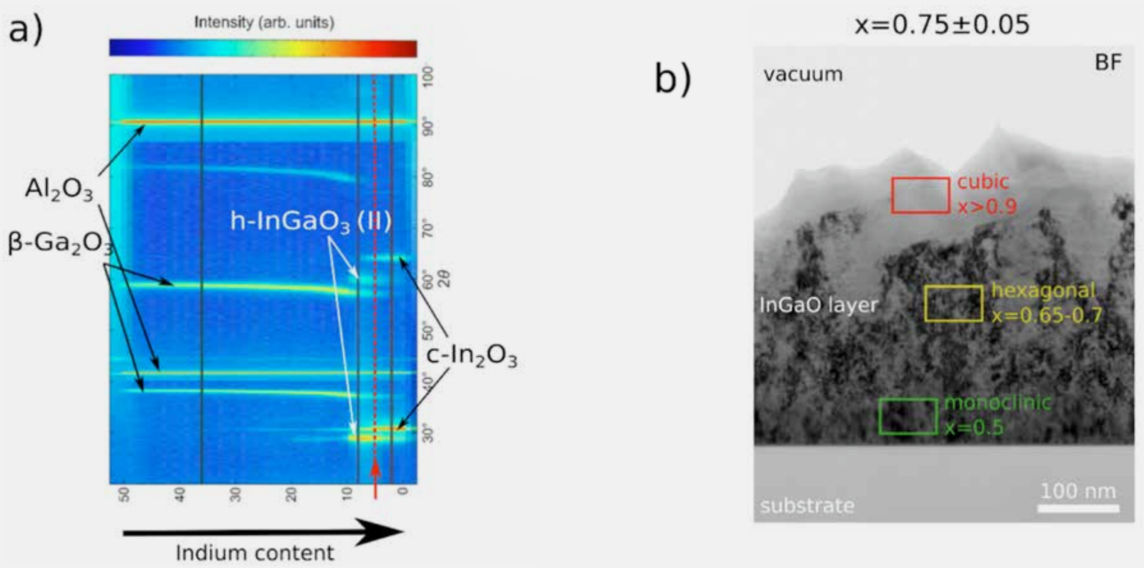


Fig. 6

a) Spatially resolved XRD ω-2θ scan of a $(In_xGa_{1-x})_2O_3/Al_2O_3$ sample grown by PLD with an indium content varying between $0<x<0.9$.

b) TEM bright field image at the position indicated by the red arrow in the XRD scan. The displayed indium contents of the respective phases are determined by a STEM-EDXS measurement.

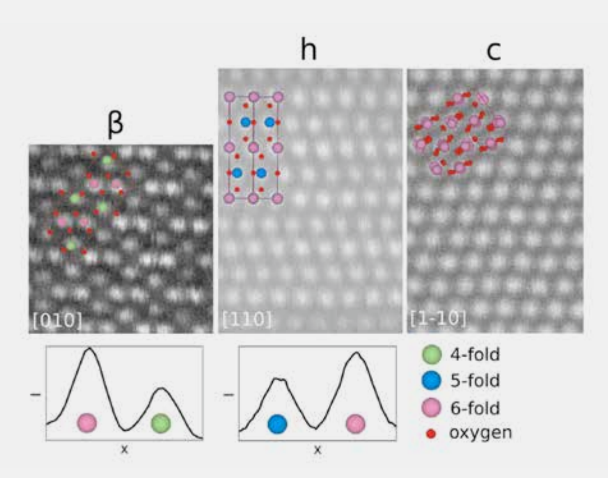


Fig. 7

Experimental high-magnification STEM-HAADF images (several images summed to enhance contrast) of the monoclinic, hexagonal and cubic $(In_xGa_{1-x})_2O_3$ alloys with overlay of stick-and-ball models. The lower plots show intensity line profiles (average of several) along the two differently coordinated atoms in the monoclinic and hexagonal lattices. The intensity difference between the 4-/5-fold and 6-fold atomic columns respectively proves the preferential incorporation of indium to the octahedral lattice sites.

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Simulation & Characterization:
Chemical & Thermodynamic Analysis

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Überblick

Service-Messungen für andere Arbeitsgruppen des IKZ beanspruchen in der Regel einen signifikanten Anteil der Arbeitskraft unserer Gruppe; naturgemäß ist die Zusammenarbeit mit der Gruppe „Oxide &Fluoride" besonders eng. Diese Zusammenarbeit betrifft thermodynamische Untersuchungen, wie die Bestimmung von Phasendiagrammen die für die Züchtung bestimmter Kristalle relevant sind, sowie die Charakterisierung von Ausgangsmaterialien durch chemische Analyse und simultane Differential-Thermoanalyse/Thermogravimetrie (DTA/TG). Chemische Analysen wurden in unserer Gruppe mit inductively coupled plasma - optical emission spectrometry (ICP-OES) bis zu seinem Renteneintritt Ende 2017 durch Dr. Rainer Bertram durchgeführt. Die Weiterführung dieser Analysenmethode am IKZ ist geplant.

Der folgende Beitrag stellt einige der Ergebnisse vor, die von Max Schmidt (Master-Abschluss Dezember 2017) erzielt wurden.

Overview

Service measurements for other IKZ groups represent a significant work share of our group; it is natural that the collaboration is especially close with the "Oxides & Fluorides" group. This collaboration includes thermodynamic investigations (such as the determination of phase diagrams relevant for the growth of specific crystals) as well as the characterization of starting materials by chemical analysis and by simultaneous differential thermal analysis/thermogravimetry (DTA/TG). In our group, Dr. Rainer Bertram performed chemical analysis with inductively coupled plasma - optical emission spectrometry (ICP-OES). He has retired by the end of 2017, and the re-installation of ICP-OES at IKZ is now under discussion.

In this contribution, we will present some of the results of the master student Max Schmidt who obtained his degree in December 2017.

Results

The chemical substance $CaTiO_3$ is found occasionally in nature as the mineral perovskite and represents the archetype for the perovskite crystal structure type. Typically this structure type is described as cubic, and it is interesting to note that for the mineral perovskite itself the unit cell is orthorhombically distorted to space group Pbnm. $CaTiO_3$ is cubic only at high temperatures; in this cubic phase Ti^{4+} sits in the center of an oxygen octahedron, and Ca^{2+} is placed in the center of an oxygen cuboctahedron. The cubic structure is stable from the melting point $T_f = (2220 \pm 20)$ °C down to ca. 1350 °C where a tetragonally distorted structure is formed. This tetragonal structure transforms at ca. 1260 °C to the orthorhombic Pbnm structure which is stable down to room temperature.

At least one of the cubic/tetragonal or tetragonal/orthorhombic phase transitions is disruptive. Hence crystal growth of $CaTiO_3$ crystals from the melt leads to twinned or even cracked crystals; in contrast to $SrTiO_3$ that is a cubic perovskite from the melting point down to room temperature and can be grown from the melt in high quality [1]. Disruptive phase transitions during cooling can be bypassed by growing crystals not from stoichiometric melts but from melt solutions with lower liquidus temperatures, compared to the pure substance. A variety of typical melt solvents such as KF, B_2O_3, PbF_2, $CaCl_2$, $BaCl_2$, CaF_2, CsF, MoO_3 were described in the literature as possible solvents for $CaTiO_3$ [2,3]. None of them allowed the growth of crystals with good quality in significant size beyond a few millimeters so far.

We could show that the treatment of solvent and solute as independent substances is an inaccurate simplification that neglects dissociation to ions upon melting and possible reorganization to new compounds upon cooling, such as typical for reciprocal salt pairs [4]. In the case of the common melt solvent potassium fluoride [3], the exchange reaction

$$CaTiO_3 + 2\,KF \rightleftarrows CaF_2 + K_2TiO_3 \tag{1}$$

leads to the formation of calcium fluoride that forms with excess KF via

$$CaF_2 + KF \rightleftarrows KCaF_3 \tag{2}$$

a double fluoride which was shown to exist in the melt. The drawback is mainly, that reaction (1) reduces the small $CaTiO_3$ assay in the system, which limits the efficiency of the growth process. Similar problems arise from most other solvents that introduce “foreign” ions into the melt solution.

It seems a straightforward solution of this problem to use solvents that do not contain any foreign ions. Recently this was demonstrated for the growth of $SrTiO_3$ that could be grown with improved quality (resulting from lower thermal gradients) from a $SrTiO_3$-TiO_2 melt solution [5]. Unfortunately, in the analogous pseudobinary system $CaTiO_3$-TiO_2 the eutectic temperature is at 1460 °C, which is significantly higher than the temperatures of both cubic/tetragonal and tetragonal/orthorhombic transitions of $CaTiO_3$ that were mentioned in the beginning. Consequently, with pseudobinary melt solutions of $CaTiO_3$ and TiO_2 crystal growth cannot be performed below these critical transitions.

Calciumfluoride is a well-known solvent for flux growth, which is also often used in metallurgy for better slag formation. Its benefit is that it introduces at least no foreign cations to $CaTiO_3$. Hillert investigated already in the 1960s mixtures of CaF_2 and TiO_2 and published a phase diagram that shows a miscibility gap in the melt near the CaF_2 side [6]. We could demonstrate that Hillert’s latter assumption was wrong: the miscibility gap exists, but a “rutile+melt” phase field below the “2 melts” field opens to the TiO_2 side instead. This difference has severe consequences for the intended application as melts solvent, because the diagram in Fig. 1 proposes rutile as a primary crystallizing phase for the largest part of the diagram, which agrees with our observations. Indeed, rutile (TiO_2) needles were found in all DTA samples with $0.4 \leq x \leq 0.8$ (x : molar fraction of CaF_2). Hillert’s diagram instead proposes CaF_2 as a primary phase for $x > 0.43$. Generally, demixing of the melt is highly beneficial for melt solution growth, because then the system remains totally molten over an extended concentration range at comparably lower temperatures. If the monotectic demixing would not occur, a TiO_2 liquidus close to the red dashed line in Fig. 1 should be expected. Melting the whole system would then require such high temperatures, that severe evaporation must be expected.

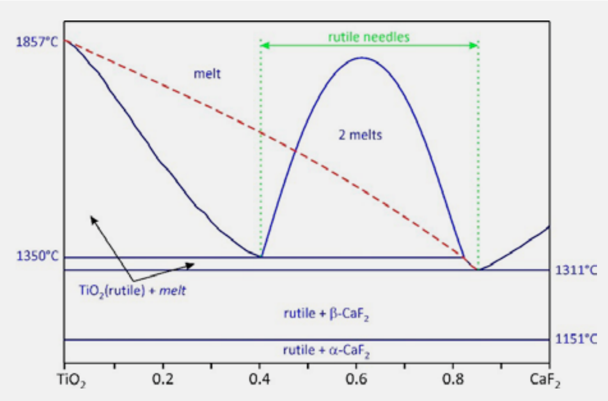

Fig. 1

Blue lines: Re-determined system CaF_2-TiO_2 [7]. The dashed red line corresponds to the hypothetical rutile liquidus if demixing of the melt would not occur. The range where rutile needles are observed in DTA samples is marked.

The new results from Fig. 1 were combined with new experimental results for the other rim systems $CaTiO_3$-TiO_2 and $CaTiO_3$-CaF_2, and additional measurements for ternary compositions. Together with literature data for the few pure stoichiometric solid phases, a thermodynamic assessment of the system was possible the introducing binary excess energy parameters for the ternary melt. The results can be presented as a projection onto the liquidus of the concentration triangle which is shown in Fig. 2.

This ternary diagram makes obvious that from solely binary systems of $CaTiO_3$ with TiO_2 or CaF_2, respectively, always cubic $CaTiO_3$ crystallizes – with the consequence that the series of critical phase transitions is performed upon cooling. In the gray region, however, the liquidus surface is significantly lower, and below the cubic/tetragonal transition. In a very small region around the ternary eutectic point, the liquidus even falls below the final tetragonal/orthorhombic transition.

First unseeded growth experiments resulted in millimeter-sized $CaTiO_3$ crystals that were optically clear and showed no signs of twinning, which is a satisfying starting point for further growth processes. These results were published recently [8].

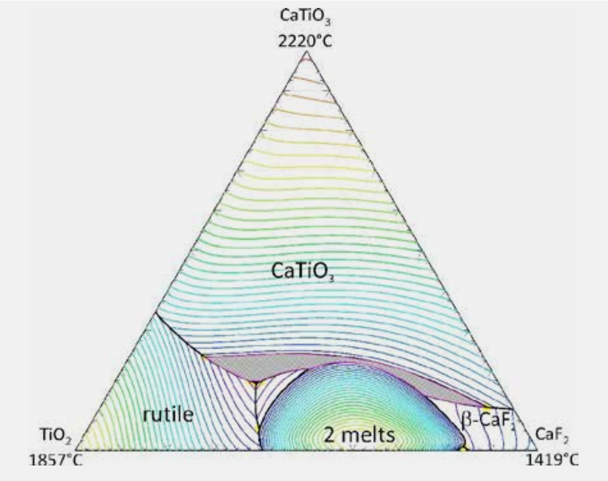

Fig. 2
Thermodynamic assessment of the $CaTiO_3$-CaF_2-TiO_2 system. $CaTiO_3$ crystallizes from a large concentration range on the top cubic, and from the gray region tetragonal.
Close to ternary eutectic point a small region with crystallization of the orthorhombic phase exists. Isotherms: 20 K.

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Simulation & Characterization:
Crystal Machining

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Überblick

Die Arbeit der Gruppe Kristallbearbeitung konzentriert sich vorrangig auf folgende Aufgaben:

- *Probenpräparation für die routinemäßige züchtungsbegleitende Diagnostik der im Haus gezüchteten Kristalle mit vielseitigen Anforderungen an die Probengeometrie und an die Oberflächenqualität, resultierend aus dem breiten Spektrum an zu bearbeitenden Materialien mit unterschiedlichen mechanischen Eigenschaften und Dimensionen.*
- *Herstellung von kristallographisch genau orientierten Kristallkeimen und Substraten für den Einsatz in der Kristallzüchtung. Vor allem die Präparation von epi-ready Substraten für die Epitaxie wird zunehmend wichtiger auch bezüglich neuer Materialien und Anforderungen.*
- *Entwicklung von Schneid- und Oberflächenpräparationstechnologien und -vorschriften, vor allem für neue am Institut gezüchtete Kristallmaterialien, um auch hierbei den Anforderungen der Züchtung und der Materialdiagnostik gerecht zu werden.*
- *Service- und Forschungsaufträge für die Industrie, für Hochschulen und außeruniversitäre Forschungseinrichtungen, die von der Lieferung bearbeiteter Proben bis hin zur Entwicklung von Technologien und die Erarbeitung der damit verbundenen Dokumentationen reichen.*

Die Präparation von Halbleitermaterialien oder von Kristallen für optische Anwendungen, für die Diagnostik oder für den Einsatz als Substrate für die Epitaxie erfordert meist eine hohe Präzision in der Bearbeitung. Unabhängig von Material oder Verwendungszweck durchläuft jede Probe bzw. jeder Wafer bis zur Fertigstellung verschiedene Arbeitsschritte wie Schneiden, Formatieren und Oberflächenpräparation mittels unterschiedlicher Schleif-, Läpp- und Polierprozesse. Die dazu in unserer Gruppe zur Verfügung stehenden Methoden umfassen:

- *röntgenografisches Orientieren von Kristallen (in Zusammenarbeit mit der Gruppe Physikalische Charakterisierung)*
- *Trennschleifen mit verschiedenen Verfahren (Diamantdraht- und Diamant-Innentrennsägen)*
- *Flachschleifen mit Diamantwerkzeugen*
- *Läppen mit verschiedenen Läppmitteln (Aluminiumoxid, Ceriumoxid, Siliciumcarbid, Borcarbid, Diamant) in verschiedener Korngrößen und Suspensionen*
- *mechanisches und chemo-mechanisches Polieren*
- *Oberflächencharakterisierung mittels Lichtmikroskopie, Atomkraftmikroskopie (AFM), Konfokalmikroskopie (CFM) und Rasterelektronenmikroskopie (REM)*
- *Bestimmung von Wafergeometrien und Oberflächenparametern wie Waferdicke, Durchbiegung, Ebenheit, Parallelität und Oberflächenrauigkeit*

Die vorhandene anlagentechnische Ausstattung und die hohe fachliche Kompetenz aller Mitarbeiter auf dem Gebiet der gesamten Probenpräparation ermöglichen es, kurzfristig und in hoher Qualität auf unterschiedliche Probenanforderungen zu reagieren.

Overview

The work of the group Crystal machining is focused on the following tasks:

- sample preparation for routine characterization and material diagnostics of the in-house grown crystals by manufacturing of samples with special requirements on sample geometry and surface parameters resulting from the wide spectrum of materials with different mechanical properties and sample sizes;
- preparation of crystallographically-oriented seed crystals and epi-ready substrates for the crystal growth. The latter task became particularly important for our group in regards to new materials and requirements;
- development of cutting and surface preparation technologies and instructions for new materials grown at the institute in order to meet the requirements for crystal growth and material diagnostics;
- various service for and research orders from industry, universities and other research institutes from the supply of machined samples to the development of technologies and their related documentation.

The preparation of crystals for semiconductor and optical applications, for diagnostics or as substrates for epitaxial processes requires high-precision machining. Regardless of the application or material, each sample or wafer undergoes several stages during manufacturing including formatting, slicing the wafer from the crystal, and preparing the surface using different grinding, lapping and polishing techniques. The methods used in our group are:

- crystal orientation using X-ray techniques in cooperation with the group “Physical Characterization”;
- crystal cutting and wafering by different methods (single and multi diamond wire and inner diameter diamond sawing);
- wafer grinding with diamond tools;
- wafer lapping with various abrasives (aluminum oxide, cerium oxide, silicon carbide, boron carbide, diamond) in different particle sizes and suspensions;
- mechanical and chemo-mechanical polishing;
- surface characterization by light microscopy, atomic force microscopy (AFM), confocal microscopy (CFM), and scanning electron microscopy (SEM);
- determination of standard wafer geometry and surface parameters like thickness, bow, evenness, planarity, and roughness.

The available equipment combined with the experience and competence of our staff members in the field of sample preparation enables us to achieve the varying requirements in short time and with high quality.

Results

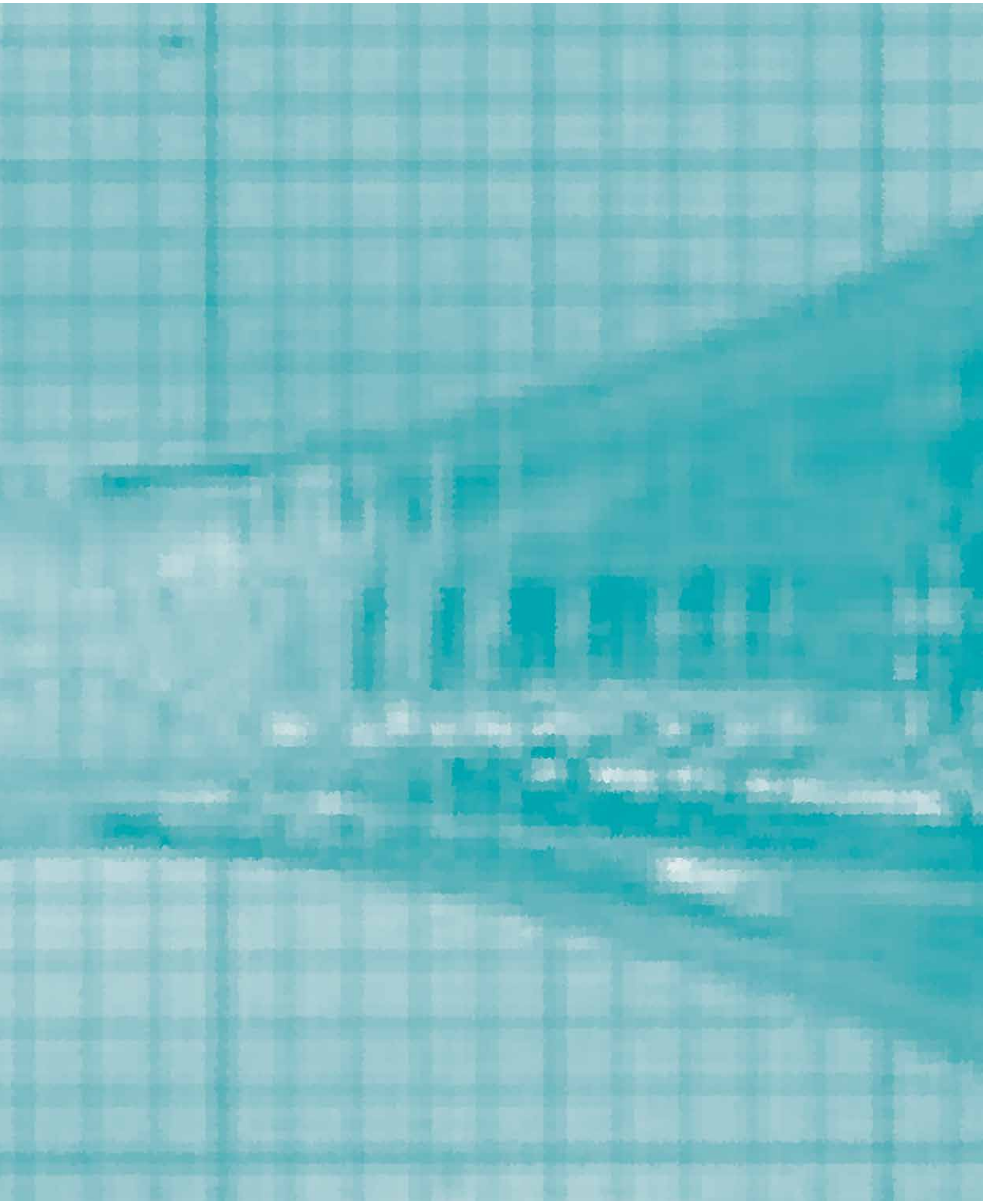
In 2017, one of our primary tasks was the development of polishing and etching methods for nitride and oxide crystals grown at our institute, with focus on Aluminum Nitride (AlN), Strontium Titanate ($SrTiO_3$) and Gallium Oxide (Ga_2O_3). The aim of this work is to prepare epi-ready surfaces for epitaxial growth using a suitable chemo-mechanical polishing (CMP) method including a high-quality mechanical pre-polishing.

CMP is a process of polishing and planarization of wafer surfaces with slurries containing abrasive particles and reactive chemicals. It represents the final step in wafer preparation. The essential tasks of CMP are the removal of scratches, surface and subsurface damages caused by the precedent mechanical polishing as well as the reduction of residual roughness and unevenness of the surface without leaving disturbing residual surface layers. A basic requirement for successful CMP is a high-quality mechanical pre-polish because CMP should remove only few microns of the material, otherwise further geometric restraints cannot be maintained.

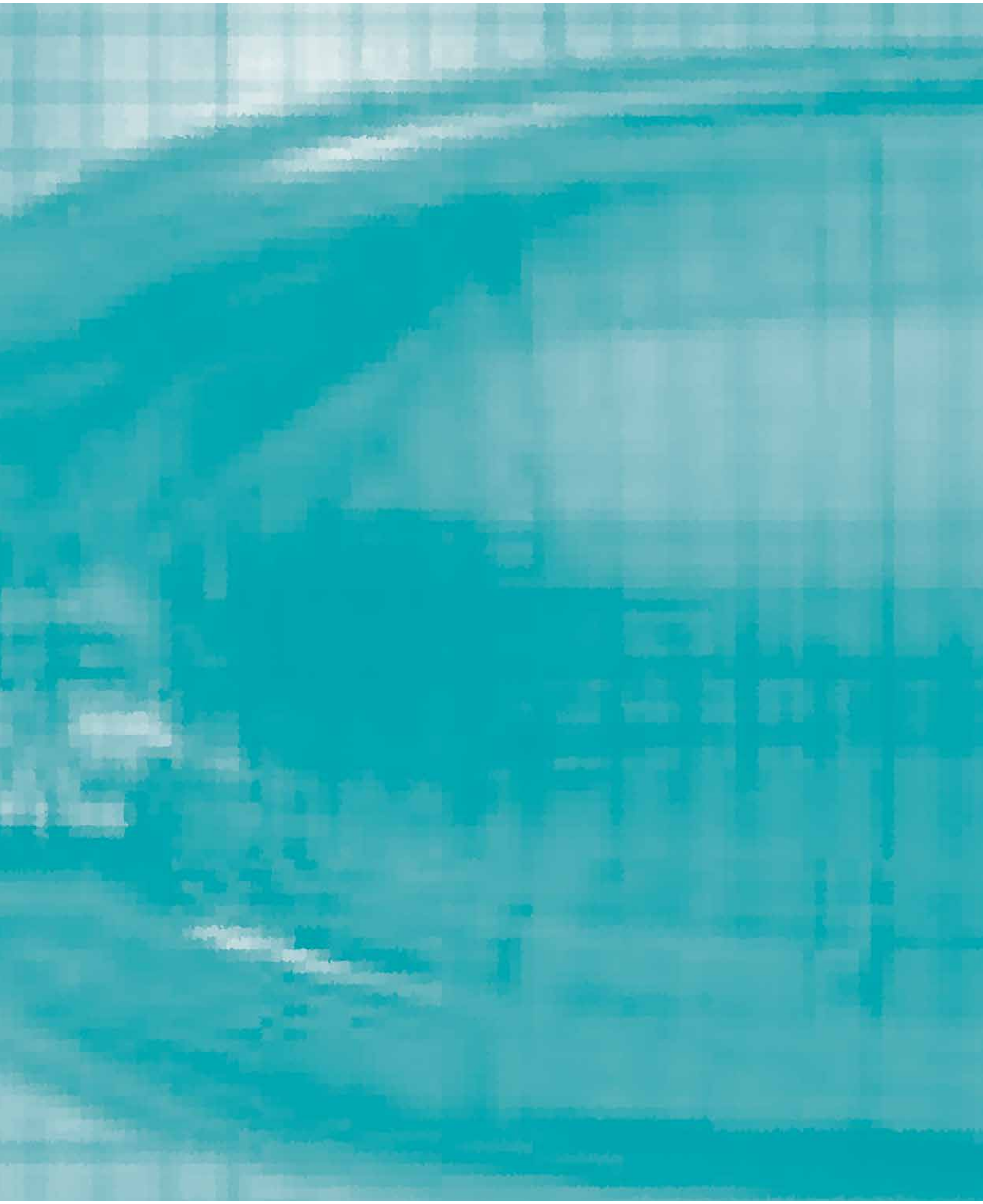
In the last years, the experiments focused on the polishing of Al-polar AlN surfaces have already been successful. As a result, we could achieve scratch-free Al-polar surfaces of high quality with a roughness value of 0.04 nm (root mean square, RMS) over large areas. To prove whether the surface has epi-ready quality, these samples were tested as substrates for homo-epitaxial layer deposition. In the last experiment, a good surface quality was obtained, which suggests an excellent CMP. Based on these results and depending on the availability of samples, the mechanical and chemo-mechanical polishing of Al-polar surfaces will be optimized and also applied to other planes of AlN crystals. First experiments with m-plains were carried out.

Another ambitious challenge was to develop a preparation technology for some oxide crystals to achieve an epi-ready surface. Different experiments on polishing using colloidal silica slurries, aluminium oxide, and cerium oxide were performed with varying process parameters like pressure, velocity of plate- and carrier rotation, polishing time, the mixture of polishing fluid, flow rate of the slurry and the type of polishing pad. In these experiments, we found that the pH-value of the polishing slurry is a parameter which should not be underestimated. It becomes apparent that polishing works both in alkaline and acidic slurries, but it has significant influence at the polishing time. First results show surfaces without scratches, and no significant edge roll off. We plan to continue work in this field in the future.

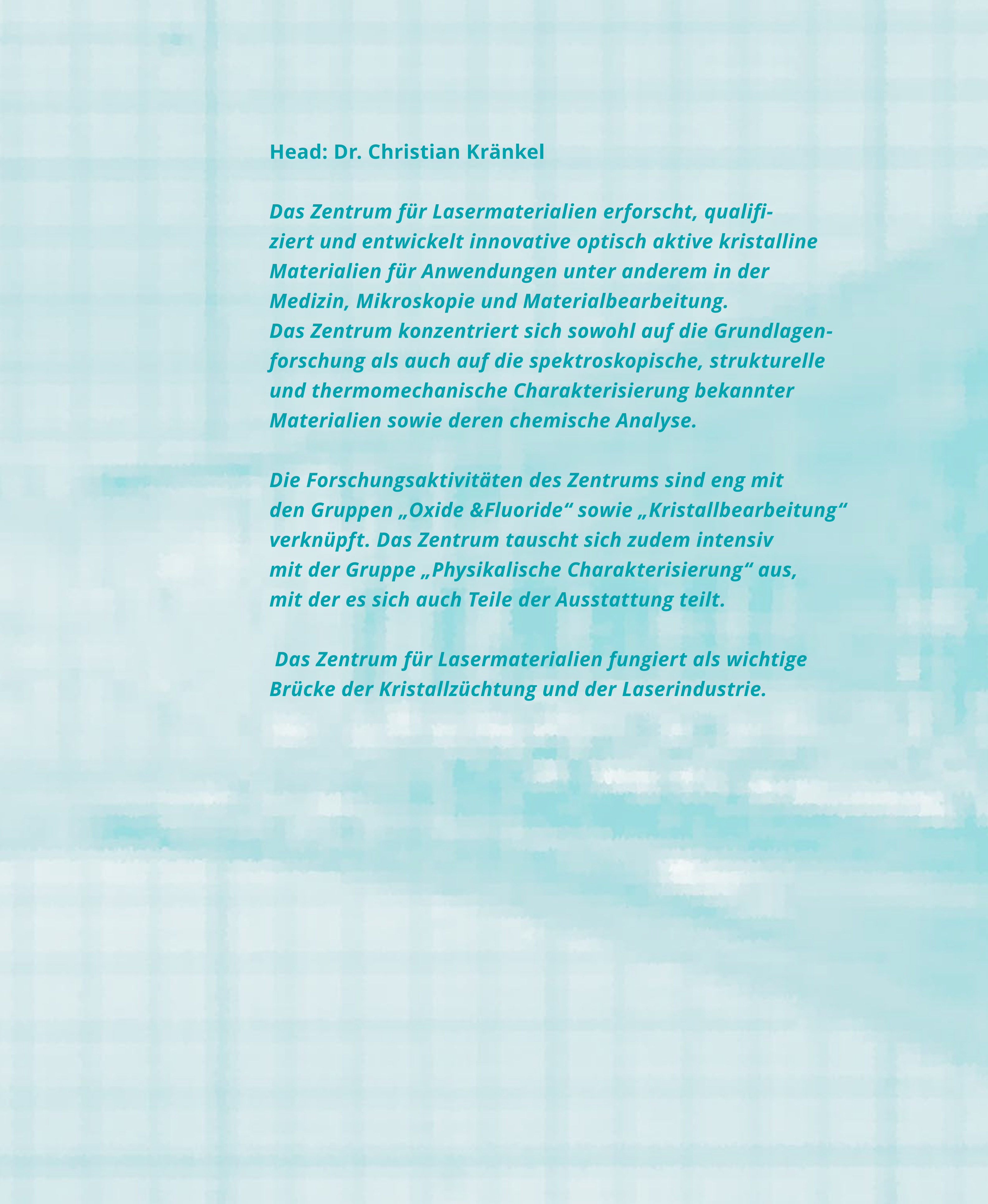
Zentrum für Lasermaterialien



Center for Laser Materials



Zentrum für Lasermaterialien



Head: Dr. Christian Kränkel

*Das Zentrum für Lasermaterialien erforscht, qualifiziert und entwickelt innovative optisch aktive kristalline Materialien für Anwendungen unter anderem in der Medizin, Mikroskopie und Materialbearbeitung.
Das Zentrum konzentriert sich sowohl auf die Grundlagenforschung als auch auf die spektroskopische, strukturelle und thermomechanische Charakterisierung bekannter Materialien sowie deren chemische Analyse.*

Die Forschungsaktivitäten des Zentrums sind eng mit den Gruppen „Oxide &Fluoride“ sowie „Kristallbearbeitung“ verknüpft. Das Zentrum tauscht sich zudem intensiv mit der Gruppe „Physikalische Charakterisierung“ aus, mit der es sich auch Teile der Ausstattung teilt.

Das Zentrum für Lasermaterialien fungiert als wichtige Brücke der Kristallzüchtung und der Laserindustrie.

Center for Laser Materials

The Center for Laser Materials explores, qualifies and develops innovative optically active crystalline materials for applications in medicine, microscopy, materials processing and others. The Center focuses on both fundamental research and spectroscopic, structural and thermomechanical characterization of known materials as well as their chemical analysis.

The research activities of the Center intertwine with the “Oxides & Fluorides”, as well as with the “Crystal Machining” groups. The Center also intensively shares knowledge and equipment with the “Physical Characterization” group.

The Center for Laser Materials acts as an important bridge between the crystal growth and the laser industry.

Center for Laser Materials

Head Dr. Christian Kränkel
Team E. Castellano-Hernández

Überblick

Das Leibniz-Institut für Kristallzüchtung hat im August 2016 das neue Zentrum für Lasermaterialien (ZLM) gegründet. Die Gründung wurde ermöglicht durch das vom BMBF unter Projektaufsicht des VDI geförderte Projekt „EQuiLa – Erforschung und Qualifizierung innovativer Lasermaterialien und -kristalle), welches in enger Zusammenarbeit mit dem Ferdinand-Braun Institut für Höchstfrequenztechnik (FBH) in Berlin durchgeführt wird. Diese Zusammenarbeit schafft ein einzigartiges Forschungsumfeld, welches Forschung entlang der gesamten Wertschöpfungskette von Lasermaterialien von der Züchtung und Konfektionierung von kristallinen Halbleiter- und Isolator-Materialien für Pumpdioden bzw. Verstärkerkristalle über deren Charakterisierung bis hin zur Auslegung und dem Aufbau diodengepumpten Festkörperlasern abbildet.

In dieser Zusammenarbeit widmet sich die Forschung am ZLM der Züchtung und Charakterisierung von Verstärkerkristallen und anderen optischen Materialien für Laseranwendungen. Außer der Erforschung neuartiger Lasermaterialien ist ein weiterer wichtiger Aspekt der Forschung am ZLM die Bereitstellung und Qualifizierung von optischen Materialien als Service für Anwender in Forschung und Industrie.

Im März 2017 konnte mit Dr. habil. Christian Kränkel ein auf diesem Gebiet ausgewiesener Experte für die Leitung des ZLM am IKZ gewonnen werden. Nachdem die Labore zunächst völlig neu ausgestattet wurden, widmeten er und sein Team sich zunächst den im Projekt EQuiLa zu erreichenden Forschungszielen. Diese gliedern sich in drei Bereiche: Im ersten Arbeitspaket werden Terbium-dotierte Perowskit-Kristalle auf ihre Eignung für sichtbare Lasertätigkeit hin untersucht. Zu diesem Zweck wurden in enger Zusammenarbeit mit der Arbeitsgruppe „Oxide & Fluoride“ am IKZ verschiedene Tb-dotierte Perowskit-Kristalle gezüchtet und hinsichtlich ihrer laserrelevanten Eigenschaften charakterisiert. Ein weiteres Arbeitspaket widmet sich der Züchtung von Sesquioxid-Kristallen in einem optischen Zonenschmelz-Verfahren (engl. Optical floating zone, OFZ). Aufgrund ihrer hohen Schmelzpunkte stellt die Züchtung dieser Materialien hohe Anforderungen an die verwendeten Tiegel- und Isolationsmaterialien, die mit hohen Kosten verbunden sind. Aus diesem Grund gelang es bisher nicht, tiegelbasierte Züchtungsverfahren im kommerziellen Bereich zu etablieren. Die tiegelfreie OFZ-Züchtung stellt einen vielversprechenden Ansatz dar, um diese Problematik zu umgehen.

Im letzten Arbeitspaket soll der „one-stop-agency“ Charakter des ZLM in Laserangelegenheiten dargelegt werden, indem ein dort diodengepumpter Cr:LiCAF-Laserresonator unter Verwendung einer am FBH hergestellten rot emittierenden InGaP-basierten Laserdiode als Pumpquelle und einem am IKZ gezüchteten Laserkristalle ausgelegt und aufgebaut wird.

Overview

The Leibniz Institut für Kristallzüchtung established the new Zentrum für Lasermaterialien (Center for Laser Materials) in August 2016. The initiating project “EQuiLa” – “Erforschung und Qualifizierung innovativer Lasermaterialien und –kristalle” (research and qualification of innovative laser materials and crystals) is funded by the BMBF. IKZ conducts the project in close collaboration with the Ferdinand-Braun Institut für Höchstfrequenztechnik (FBH) in Berlin. This collaboration creates a unique research environment, which enables research along the whole value chain of laser materials: from the growth and fabrication of semiconductor and insulator crystal gain materials for pump laser diodes and solid-state laser gain crystals, respectively, over the characterization to the layout and assembly of diode-pumped solid-state lasers.

To this end, the research at the ZLM is dedicated to the characterization and qualification of laser crystals and other optical materials with respect to laser applications. Besides research on new materials, the ZLM also performs a service job for research groups and industry users by supplying and qualifying optical materials.

In March 2017 the IKZ hired Dr. habil. Christian Kränkel as the head of the ZLM. Since then he and his team are setting up the laboratories with the required equipment and pursue the goals of the 18 month EQuiLa project.

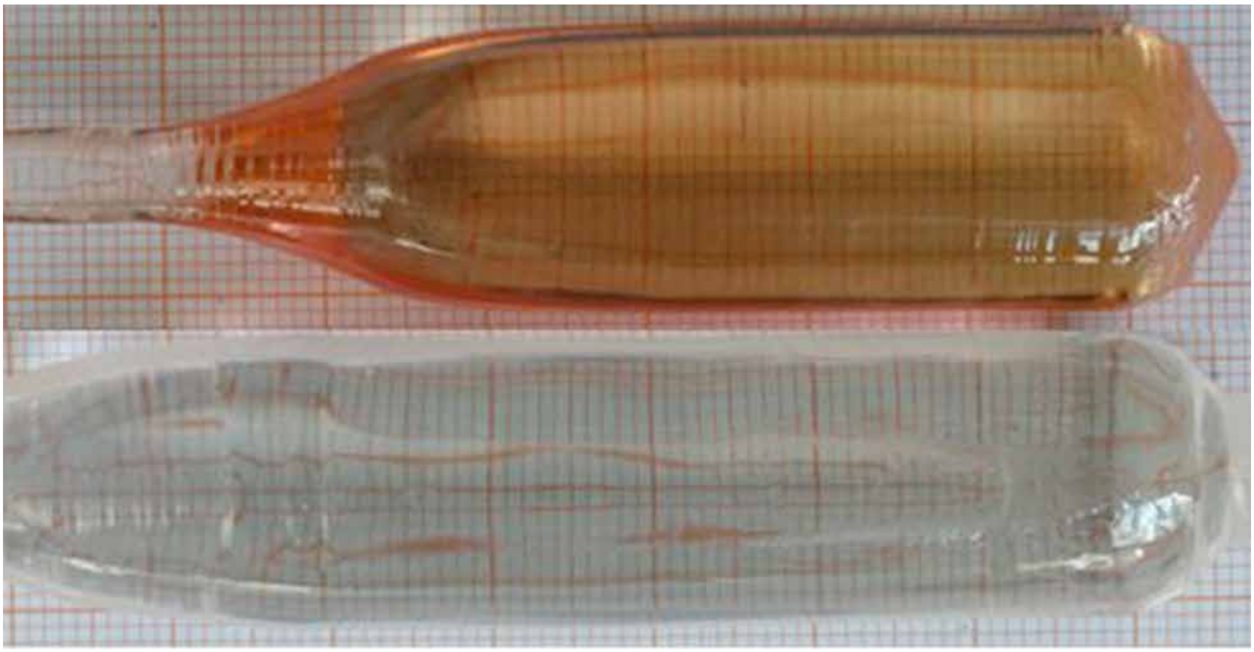


Fig. 1
Growth results of Tb(5%):YAP (top) and undoped YAP (bottom) shown on millimeter paper.

The first main research topic within the project is the investigation of novel host materials for visible lasers, using, in particular, the trivalent rare earth terbium (Tb^{+3}) as the active ion. In close collaboration with the "Oxides & Fluorides" group, the terbium-doped perovskite crystals were grown at the IKZ and their laser-related spectroscopic properties were characterised. The second objective is to establish the optical floating zone growth (OFZ) technique for the fabrication of sesquioxide crystals (lutetia, scandia, and yttria) doped with the trivalent rare earth ion erbium (Er^{+3}) for lasers in the mid-infrared spectral range.

Due to the high melting points of these materials, the existing growth methods require highly expensive rhenium crucibles and insulation materials, and therefore are not suitable for commercialization. The crucible-free OFZ growth can provide a viable solution. Finally, the project aims to demonstrate the one-stop-agency character of the ZLM by setting up a diode-pumped Cr:LiCAF laser utilizing a gain crystal grown at the IKZ and an InGaP-based red-emitting laser diode grown and assembled at the FBH.

Results

In 2017, the work at the ZLM was mainly devoted to the installation of the laboratories and the acquisition of the equipment required for the scientific tasks. Meanwhile, the main laboratory for spectroscopic investigations and laser tests is nearly fully in operation. Further scientific instruments such as crystal growth facilities and a transmission spectrometer were integrated into the existing laboratories for oxide and fluoride crystal growth and physical characterization.

Oxide and fluoride crystals for visible lasers

Besides the challenging task of setting up new laboratories, there was some significant scientific progress in the research topics of the ZLM in the past year, in particular, in the research on Tb^{3+}-doped materials for visible lasers. In ongoing experiments, we improved the performance of Tb^{3+}-doped fluoride lasers with emission in the yellow and green spectral range and realized the first continuous-wave lasers based on this ion with tuneable wavelength. In cooperation with the Universität Hamburg we obtained wavelength tuning ranges of 7 nm and 10 nm, respectively, in the green spectral range with Tb^{3+}:$LiLuF_4$ and Tb^{3+}/Na^+:CaF_2. Moreover, in experiments with TbF_3 we proved, that even this 'fully-doped' stoichiometric crystal is suitable for lasing. It opens the way for further improvement of the performance using very high doping concentrations in other host matrices [2].

Furthermore, four Tb^{3+}-doped oxide crystals for visible lasers with the compositions $Tb_xY_{1-x}AlO_3$ (Tb:YAP) where x = 0, 0.05, 0.1, and 0.2 were grown. These crystals are more than 50 mm long and have diameters of about 20 mm (see Fig. 1). We currently characterize the spectroscopic properties of these crystals to determine whether they are suitable for visible laser applications. Tb^{3+}-doped oxide crystals are potentially more suitable for high-power laser operation than fluoride crystals mentioned above, due to better thermo-mechanical properties.

Fig. 2.

Left: Result of the first OFZ-growth attempt of Er(14%):Y_2O_3 performed in a furnace at the company SciDre. The left white part shows the un-melted sintered ceramic feeding rod, the violet part on the right is the as-grown crystal.

Right: Outcome of the OFZ-growth of undoped Lu_2O_3 utilizing a furnace of the company MaTecK.

Oxide crystals for the lasers in the mid-infrared range

The IKZ acquired a new facility for optical floating zone (OFZ) growth, however by the end of 2017 it was not in operation yet. Nevertheless, we performed initial experiments on the OFZ growth of sesquioxide crystals in collaboration with the supplier of the facility, SciDre, and the company MaTecK that uses similar equipment in commercial applications. The primary goal of these experiments was to assess the possibility to melt and grow sesquioxide crystals, which have high melting points above 2400 °C, using the crucible-free OFZ technique. Figure 2 shows the results of the initial growth attempts.

The Er^{3+}-doping causes the purple color of the Er(14%):Y_2O_3 crystal shown on the left side. The crystal exhibits several cracks due to not optimized growth parameters and a phase transition below the melting point. However, a proper choice of the growth parameters and the feeding rod composition will open the prospects for growing crack-free crystals. This assumption is further supported by the un-doped Lu_2O_3 crystal shown on the right-hand side, which exhibits an excellent crystal quality.

The dimensions of the lutetia crystal are smaller due to the smaller size of the feeding rod in the initial experiment, but the crystal is free of cracks and even mainly free of color centers.

The deficiency of oxygen is a known source of color centers, however, for crystal growth from rhenium-crucibles oxygen partial pressure has to be very low to prevent oxidation of the crucible material. Using the crucible-free OFZ-method allows to circumvent this problem and to grow even in air thus preventing the formation of color centers.

Furthermore, we established Co- and Cr-doped materials as new classes of saturable absorbers for visible lasers. Guest students from the groups of Prof. N. Kuleshov from the BTNU in Minsk, Belarus, and Prof. F. Kannari from the Keio University, Yokohama, Japan supported this work. Linear and nonlinear absorption properties of various host materials doped with these ions were analyzed in detail. Besides, we performed successful experiments on passively Q-switched laser operation at various visible laser wavelengths of Pr:YLF utilizing Co-doped spinel as a saturable absorber [1].

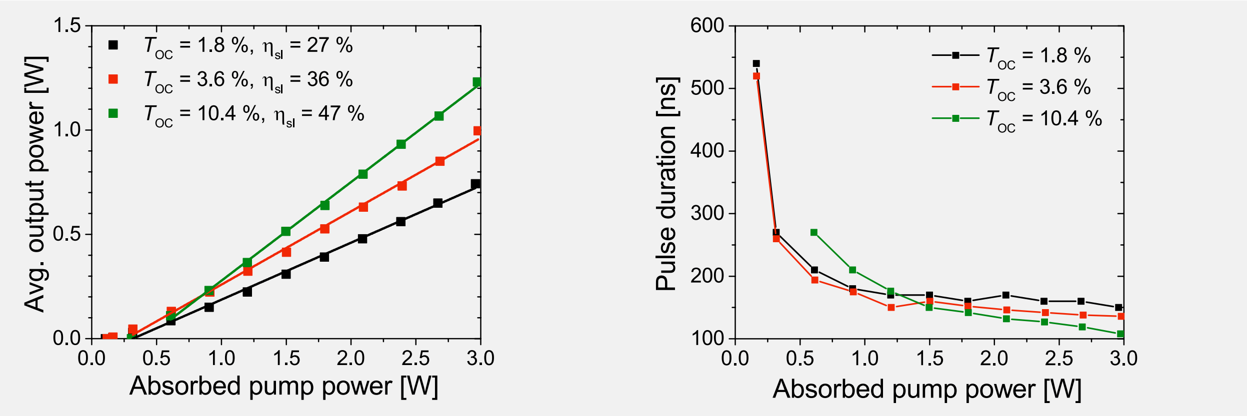


Fig. 3

Laser characteristics and pulse duration vs. pump power for a visible Pr:YLF laser Q-switched with Co^{2+}-doped spinel and operated at 640 nm.

In these experiments, we demonstrated the highest average output power for any passively Q-switched Pr^{3+}-doped laser (see Fig. 3, left). The pulse durations in the order of 100 ns (Fig. 3, right) are among the shortest obtained with this kind of laser materials. Moreover, even with the 0.75 mm thick anti-reflection coated Co:spinel passive saturable absorber into the cavity, the slope efficiency remained as high as 47%, pointing towards the low amount of non-saturable losses of this novel saturable absorber material.

Important scientific collaborations

Further research achievements are the result of ongoing collaborations with research groups all over the world. In collaborations with the ETH Zurich (Prof. U. Keller) and the University of Neuchâtel (Prof. T. Südmeyer), gain materials were supplied, which enabled an improved peak power [3] and shorter pulse durations of mode-locked thin disk lasers at 1 µm. Pulse durations as short as 35 fs [4] enabled the first application of these lasers for the generation of high harmonics at MHz repetition rates inside the cavity of such an oscillator [5].

In a further collaboration with the group of Ass. Prof. Masaki Tokurakawa at the University of Electro-Communication in Tokyo, Japan, the first Kerr lens mode-locked laser in the 2 µm wavelength range based on an inband-pumped $Tm:Sc_2O_3$ crystal was realized [6]. This result is remarkable, as, due to the larger beam diameters in long wavelength lasers and the lower nonlinear refractive index at these wavelengths, it is more difficult to achieve the required intensities to make use of the Kerr effect. In-band pumped by an Er^{3+}:fiber amplifier the laser delivered pulses as short as 166 fs with an average output power of 440 mW at a central wavelength of 2124 nm. These results motivate further research on the optimization of sesquioxide host materials in future projects at the ZLM.

A collaboration with researchers at the Shandong University in Jinan, China (Group of Ass. Prof. T. Li) we introduced graphitic carbon nitride films as saturable absorbers for lasers based on Er^{3+} as well as Ho^{3+}/Pr^{3+}-codoping in the mid-infrared spectral range around 3 µm [7, 8].

Publications

Researchers of the ZLM co-authored several publications on waveguide lasers with narrow emission bandwidth [9, 10], waveguide amplifiers [11] and the first laser action of a rare-earth doped sapphire crystal [12]. The latter was based on a Nd-doped sapphire thin film grown by pulsed laser deposition. Only due to the growth regime out of the thermal equilibrium the implementation of the large Nd^{3+}-ion into the sapphire matrix became feasible.

The results in this field are the outcome of the research performed in the previous group of C. Kränkel at the Institut für Laser-Physik at the Universität Hamburg, Germany.

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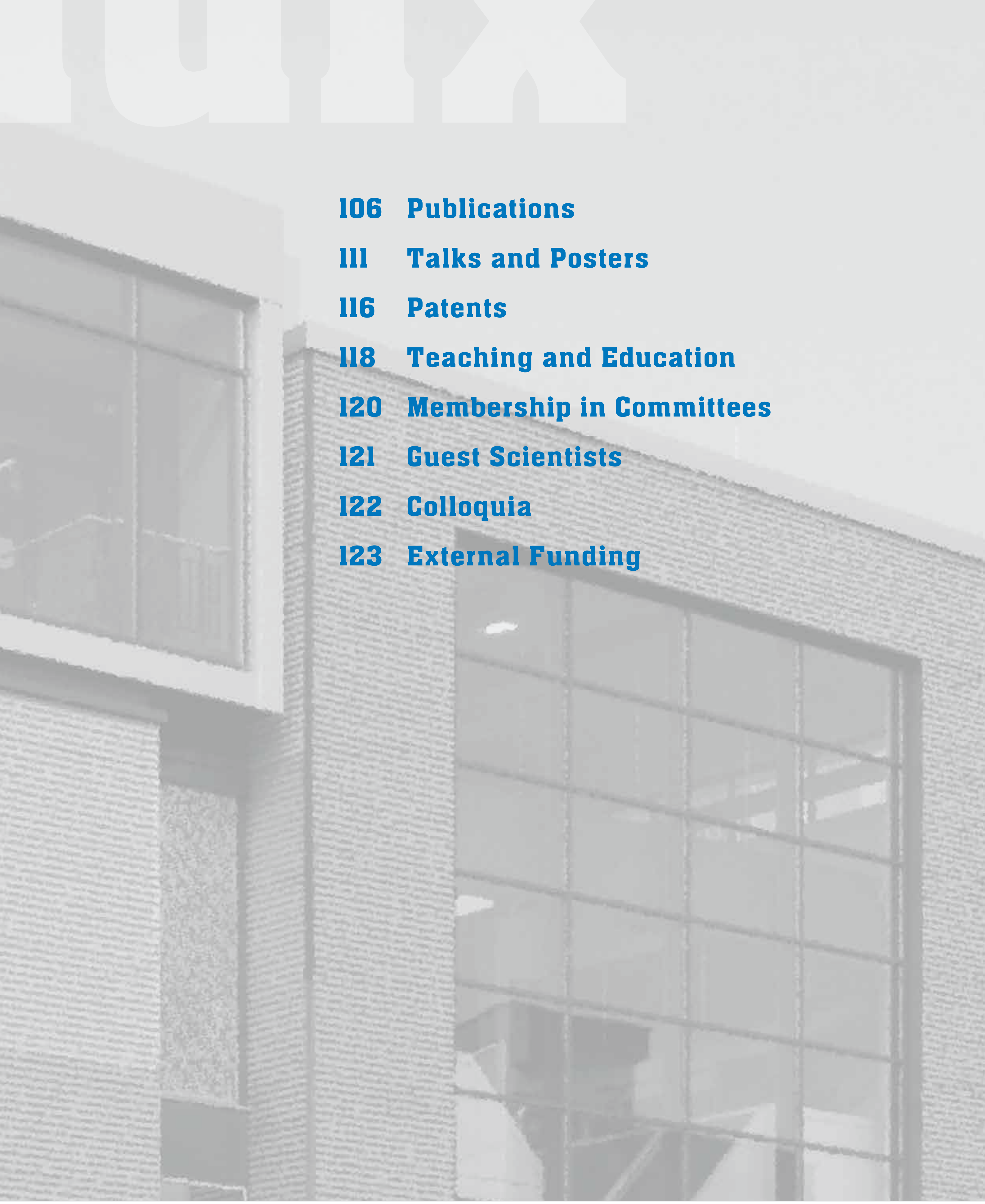

Appendix: **Publications**

Articles in books

D. Klimm; *"Thermodynamic and Kinetic Aspects of Crystal Growth"* in Handbook of Solid State Chemistry, Wiley-VCH (2017), 375-398

Articles in international peer-reviewed journals and proceedings

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Appendix: **Talks and Posters**

Invited talks at national and international conferences

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M. Albrecht; *Atomic defects studied by transmission electron microscopy*; M. Albrecht, T. Remmele, R. Schewski, T. Markurt, Z. Galazka, J. Varley, C. Van de Walle; EUROMAT 2017; Thessaloniki, Greece, August 17-22, 2017

M. Albrecht; *Strain mapping in the real and in the reciprocal space*; M. Albrecht, T. Schulz, T. Remmele, T. Markurt; Autumn School on Electron Microscopy in Materials Science, Berlin, Germany, October 9 -12, 2017

M. Bickermann; *Defects in AlN Bulk Crystal Substrates for UV LEDs and Lasers*; C. Hartmann, A. Dittmar, S. Sintonen, S. Kollowa, T. Schulz, K. Irmscher, J. Wollweber; 17. Conference on Defects-Recognition, Imaging and Physics in Semiconductors (DRIP-17); Valladolid, Spanien; October 8-12, 2017

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C. Guguschev; *Perovskite substrate crystals for strain engineering*; D. Klimm, C. Guguschev, M. Brützam, Z. Galazka, D. J. Kok, U. Juda, R. Uecker, M. Bickermann; 12th Pacific Rim Conference on Ceramic and Glass Technology (PACRIM 12); Hawaii, USA; June 21-26, 2017

K. Irmscher; *Defect related electrical and optical properties of AlN bulk crystals grown by physical vapor transport*; C. Hartmann, S. Kollowa, T. Schulz, M. Naumann, M. Pietsch, J. Wollweber, M. Albrecht, M. Bickermann; American Physical Society (APS) March Meeting; New Orleans, USA; March 13-17, 2017

K. Irmscher; *Doping and defects in ß-Ga2O3*; A. Fiedler, Z. Galazka, M. Baldini, G. Wagner, R. Schewski, M. Albrecht; 2nd International Workshop on Ga_2O_3 and Related Materials (IWGO 2017); Parma, Italy; September 12 – 15, 2017

Appendix: **Talks and Posters**

J. Janicsko-Csathy; *Ge detectors for neutrinoless double beta decay searches*; 2017 International Germanium Detector Technology Workshop, Berkeley, CA, USA; December 4 – 5, 2017

J. Janicsko-Csathy; *HPGe crystal growth and characterization: progress at IKZ*; LEGEND collaboration meeting and analysis workshop; Berkeley, CA, USA; December 6-9, 2017

F. M. Kiessling; *Possibilities of HPGe crystal production and characterization at IKZ*; GERDA/LEGEND International workshop; Gran Sasso, Italy; May 15 – 17, 2017

C. Kränkel; *Novel trends in semiconductor-laser-pumped rare-earth-doped solid-state lasers*; Optical Coatings for Laser Applications OCLA; Buchs, Switzerland; April 12, 2017

C. Kränkel; *Semiconductor laser pumped visible rare-earth doped lasers;* 6th Advanced Lasers and Photon Sources Conference ALPS'17; Yokohama, Japan; April 18-21, 2017

T. Schulz; *InGaN structure at the atomic scale - correlation to optical properties*; T. Schulz, M. Anikeeva, M. Albrecht; Leibniz-JST-Workshop "Advanced Material Sciences", Dresden, Germany, September 20–22, 2017

T. Schulz; *Structural and optical investigation of (In,Ga)N/GaN short-period superlattices*; T. Schulz, M. Anikeeva, M. Albrecht, J. Moneta, I. Gorczyca, T. Suski, M. Siekacz, P. Wolny, M. Sawicka, C. Cheze and R. Calarco; Spring School; Warsaw, Poland; February 27 - March 2, 2017

Invited seminars at national and international institutions

J. E. Boschker; *Growth of 2D materials: From fundamentals to nanoscale applications*; Max Planck Institute for solid state research, Stuttgart, Germany; May, 2017

D. Siche; *Kristallzüchtung – die unterschätzte Materialbasis in der Wertschöpfungspyramide*; Invited lecture Series Physics, BTU-Cottbus-Senftenberg; Cottbus, Germany, November 11, 2017

M. Bickermann; *On the Novel Wide Band-Gap Semiconductors AlN and β-Ga_2O_3: Bulk Crystal Growth, Homoepitaxy, Properties and Applications*; Lecture in the Group "Crystal Dynamic" of Prof. Kaikomoto at RIAM, Kyushu University; Fukuoka, Japan, November 13, 2017

C. Hartmann; *Growth of bulk AlN crystals by physical vapor transport*; Seminar at the Institute of Solid State Physic in Technische Universität Berlin; July 5, 2017

J. E. Boschker; *Growth of 2D materials: From fundamentals to nanoscale applications*; Felix-Bloch-Institut für Festkörperphysik, Universität Leipzig; Leipzig, Germany; May, 2017

C. Kränkel; *Pulsed visible lasers and other research topics at the new center for laser materials in Berlin*; Seminario di Fisica della Materia, Università di Pisa, Pisa, Italy; 2017

Oral contributions at national and international conferences

N. V. Abrosimov; *Technological peculiarities related to the growth of isotopically enriched silicon single crystals*; N. V. Abrosimov, H. Riemann; 17th Conference Gettering and Defect Engineering in Semiconductor Technology (GADEST 2017); Lopota, Georgia; October 1 – 6, 2017

R. Bansen; *Growth of silicon on glass substrates from Sn solution*; C. Ehlers, R. Bansen; BTU Graduate Research School: Cluster "FuSion" Kick-Off-Meeting; Cottbus, Germany; February 2017

M. Bickermann; *Status of AlN Bulk Crystal Growth for Use as Substrates in Deep-UV Applications*; C. Hartmann, A. Dittmar, J. Wollweber, S. Sintonen, T. Schulz, K. Irmscher, A. Knauer, M. Weyers, M. Bickermann; Compound Semiconductor Week 2017 (CSW 2017); Berlin, Germany; May 14-18, 2017

M. Bickermann; *Oxide and Nitride Single Crystals for Novel Optical and Electronic Applications*; M. Bickermann, S. Ganschow, D. Klimm, G. Tränkle; 92. Annual Meeting of the German Ceramic Society (DKG 2017); Berlin, Germany; March 19-22, 2017

M. Bickermann; *Status of AlN Bulk Crystal Growth for Use as Substrates in Deep-UV Applications*; C. Hartmann, A. Dittmar, J. Wollweber, S. Sintonen, T. Schulz, K. Irmscher, A. Knauer, M. Weyers, M. Bickermann; 5th German-Swiss Conference on Crystal Growth (DKT-2017); Freiburg im Breisgau, Germany, March 8 – 10, 2017

J. E. Boschker; *Electrical performance of phase change memory cells with textured $GeTe/Sb_2Te_3$ superlattices;* 13th meeting of the working group "Materials for Non-volatile Memories", Johannes Gutenberg-Universität; Mainz, Germany; June, 2017

Appendix: **Talks and Posters**

K. Böttcher; *Numerical Modelling of the growth of beta-Ga_2O_3 single crystals by the Czochralski method*; K. Böttcher, W. Miller, Z. Galazka, J. Schreuer; 5th German-Swiss Conference on Crystal Growth (DKT-2017); Freiburg im Breisgau, Germany; March 8 – 10, 2017

D. Braun; *Variable ferroelectric domain wall alignment in strained monoclinic $K_xNa_{1-x}NbO_3$ epitaxial films*; D. Braun, M. Schmidbauer, M. Hanke, A. Kwasniewski, J. Schwarzkopf; EMRS Fall Meeting, Warsaw, Poland; September 18-21, 2017

K. Dadzis; *High-frequency Heat Induction Modeling for a Novel Silicon Crystal Growth Method*; K. Dadzis, R. Menzel, M. Ziem, T. Turschner, H. Riemann, N. V. Abrosimov; VIII International Scientific Colloquium Modelling for Materials Processing; Riga, Latvia; September 21-22, 2017

K. Dadzis; *Upscaling of silicon crystal growth from a granulate crucible*; K. Dadzis, R. Menzel, H. Riemann, N. V. Abrosimov; DGKK-Arbeitskreis Herstellung und Charakterisierung von massiven Halbleiterkristallen; Freiberg, Germany; October 11 – 12, 2017

N. Dropka; *Towards graphite-free hot zone for directional solidification of silicon*; N. Dropka, I. Buchovska, I. Herrmann-Geppert, F. M. Kiessling, U. Degenhardt; ACCGE-21/OMVPE-18, Santa Fe, USA; July 30 – August 04, 2017

N. Dropka; *TMF optimization in VGF crystal growth of GaAs by artificial neural networks and Gaussian process models;* N. Dropka, M. Holena and Ch. Frank-Rotsch; XVIII International UIE-Congress on Electrotechnologies for Material Processing, Hannover, Germany; June 6 – 9, 2017

C. Ehlers; *In situ removal of a native oxide layer on silicon subtrates by UV-laser for epitaxial applications*; C. Ehlers, R. Bansen, D. Uebel, S. Schaller, Th. Teubner, T. Boeck; 5th German-Swiss Conference on Crystal Growth (DKT-2017); Freiburg im Breisgau, Germany; March 08-10, 2017

C. Ehlers; *UV-laser assisted desorption of a native oxide layer on silicon substrates for epitaxial processes*; BTU Graduate Research School: Status Meeting of Cluster "FuSion"; Cottbus, Germany; October, 2017

K. Eylers; *In-Ga precursor islands for CIGSe micro-concentrator solar cells*; K. Eylers, F. Ringleb, B. Heidmann, S. Levcenco, T. Unold, H. W. Klemm, G. Peschel, A. Fuhrich, Th. Teubner, T. Schmidt, M. Schmid, T. Boeck; 44th IEEE Photovoltaic Specialists Conference; Washington, DC, USA; June 2017

K. Eylers; *In-Ga precursors for CIGSe micro-concentrator solar cells;* BTU Graduate Research School: Status meeting of Cluster "FuSion"; Cottbus, Germany; October, 2017

K. Eylers; *Materials research on Cu(In,Ga)Se_2 (CIGSe) micro-concentrator solar cells*; K. Eylers; BTU Graduate Research School: Cluster "FuSion" Kick-Off-Meeting; Cottbus, Germany; February, 2017

A. Fiedler; *Photo- and Electroluminescence of chromium doped β-Ga_2O_3*; A. Fiedler, Z. Galazka, K. Irmscher; DPG Spring Meeting 2017, Dresden, Germany; March 19 – 24, 2017

A. Fiedler; *Photo- and Electroluminescence of chromium doped β-Ga_2O_3*; A. Fiedler, Z. Galazka, K. Irmscher; Transparent Conductive Oxides (TCO)-2017; Leipzig, Germany; September 18 – 22, 2017

A. Fiedler; *Photo- and Electroluminescence of chromium doped β-Ga_2O_3*; A. Fiedler, Z. Galazka, K. Irmscher; 2nd International Workshop on Gallium Oxide and Related Materials (IWGO); Parma, Italy; September 12 – 15, 2017

Ch. Frank-Rotsch; *Application of heater magnet module for improved crystal growth of semiconductors*; I. Buchovska, N. Dropka, R. Zwierz, P. Rudolph, F. M. Kiessling; 21st American Conference on Crystal Growth and Epitaxy (AACGE-21); Santa Fe, New Mexiko, USA; July 30 – August 4, 2017

C. Hartmann; *Impurity management of AlN bulk crystals grown by physical vapor transport*; 10th International Workshop on Bulk Nitride Semiconductors (IWBNS-X); Espoo, Finnland; September 18 – 22, 2017

S. Kayser; *Computational Simulations of the Lateral-Photovoltage-Scanning-Method*; S. Kayser, A. Lüdge, K. Böttcher; VIII International Scientific Colloquium Modelling for Materials Processing; Riga, Latvia; September 21 – 22, 2017

S. Kayser; *Investigation of the local resolution of the LPS-Method with respect to the doping concentration using Finite-Volume-Simulations;* S. Kayser, A. Lüdge, K. Böttcher; DGKK-Arbeitskreis: Herstellung und Charakterisierung von massiven Halbleiterkristallen; Freiberg, Germany, October 11 – 12, 2017

C. Kränkel; *fs-laser inscribed waveguides in crystals for compact and versatile lasers*; Laser Ignition Summer School 2017, Brasov, Romania; July 19-22, 2017

Appendix: **Talks and Posters**

C. Kränkel; *Growth of laser crystals and its influence on the laser performance*; Laser Ignition Summer School 2017, Brasov, Romania; July 19-22, 2017

C. Kränkel; *Zentrum für Lasermaterialen at the Leibniz Institut für Kristallzüchtung*; ZLM Kick-off meeting and laser materials workshop 2017, Berlin, Germany; 2017

C. Kränkel; *GHz Mode-Locked Yb:YAG channel waveguide Lasers*; S. Y. Choi, T. Calmano, F. Rotermund, C. J. Saraceno, and C. Kränkel; Advanced Solid-State Lasers Conference 2017; Nagoya, Japan, October 1-5, 2017

C. Kränkel; *Novel crystals for tailored solid state lasers*; Photonic-Tage Berlin-Brandenburg, Workshop „Innovative optical components and technical optics“; Berlin, Germany; October 18, 2017

C. Kränkel; *Progress in laser crystal fabrication*; Advanced Solid-State Lasers Conference 2017, panel discussion on “Advanced solid-state laser materials and devices”; Nagoya, Japan; October 1-5, 2017

F. Lange; *MBE growth of Si, Ge and* Si_xGe_{1-x} *nanowires and modeling of synthesis conditions for multi-component compounds for thermoelectrics;* BTU Winter School 2017 on Characterization of micro- and nano-materials: Functional materials and analytics; Cottbus, Germany; March, 2017

F. Lange; *MBE growth of Si, Ge and* Si_xGe_{1-x} *nanowires*; BTU Graduate Research School: Workshop of Cluster “FuSion”; Dresden, Germany; July, 2017

F. Lange; *MBE growth of Si, Ge and* Si_xGe_{1-x} *nanowires*; BTU Graduate Research School: Status meeting of Cluster “FuSion”; Cottbus, Germany; October, 2017

F. Lange; *MBE growth of Si, Ge and* Si_xGe_{1-x} *nanowires*; F. Lange; BTU Graduate Research School: Cluster “FuSion” Kick-Off-Meeting; Cottbus, Germany; February, 2017

T. Markurt; *Lattice site analysis of cerium dopants in calcium scandate crystals*; T. Markurt, C. Guguschev, J. Philippen, M. Albrecht; 20th Microscopy of Semiconducting Materials – MSM XX, Oxford, UK; April 09 – 13, 2017

J. Schwarzkopf; *Domain engineering in* $K_xNa_{1-x}NbO_3$ *thin films by the application of anisotropic lattice strain*; J. Schwarzkopf, L. v. Helden, D. Braun, Y. Dai, R. Wördenweber, M. Hanke, M. Schmidbauer; TO-BE Spring Meeting 2017; Neumünster Abbey, Luxembourg; April 3-5, 2017

L. von Helden; *Monoclinic Superdomains in Ferroelectric* $K_{0.7}Na_{0.3}NbO_3$ *Thin Films on* $TbScO_3$; M. Schmidbauer, C. Feldt, D. Braun, M. Hanke, Y. Dai, R. Wördenweber, J. Schwarzkopf; DPG Spring Meeting 2017; Dresden, Germany; March 19-24, 2017

L. von Helden; *Monoclinic Domains in Strained, Ferroelectric* $K_{0.7}Na_{0.3}NbO_3$ *Thin Films on* $TbScO_3$ *– First Steps to Surface Acoustic Wave Sensors*; M. Schmidbauer, C. Feldt, D. Braun, M. Hanke, S. Liang, R. Wördenweber, J. Schwarzkopf; European Materials Research Society Fall Meeting; Warsaw, Poland; September, 2017

N. Wolff; *Investigations on the growth of Delafossite substrate crystals*; N. Wolff, D. Siche, D. Klimm; 6th German-French Workshop on Oxide, Dielectric, and Laser Crystals; Bordeaux, France; September 14 – 15, 2017

N. Wolff; *Untersuchungen zur Züchtung von Delafossit-Substratkristallen*; Naturwissenschaftstag Lausitz, Cottbus, June 6, 2017

Posters presentations at national and international conferences

C. Guguschev, S. Ganschow, D. Klimm, Z. Galazka, M. Brützam, I. M. Schulze-Jonack, R. Bertram, M. Bickermann*; Recent progress in perovskite crystal growth at the IKZ*; 6th German-French Workshop on Oxide, Dielectric, and Laser Crystals (WODIL 2017); Bordeaux, France; September 14-15, 2017

Z. Galazka, R. Uecker, D. Klimm, S. Ganschow, K. Irmscher, R. Bertram, A. Kwasniewski, R. Schewski, A. Fiedler, M. Albrecht, M. Pietsch, C. Guguschev, M. Bickermann; *Bulk single crystals of transparent semiconducting oxides*; 6. German-French Workshop on Oxide, Dielectric, and Laser Crystals (WODIL 2017); Bordeaux, France; Sep 14-15, 2017

N. Wolff, D. Klimm, C. Guguschev, D. Siche; *Thermodynamic studies of* $CuAlO_2$ *– a potential transparent p-type oxidic semiconductor*; 5th German-Swiss Conference on Crystal Growth; Freiburg, Deutschland; March 08 – 10, 2017

Appendix: **Talks and Posters**

R. Zwierz, N. Dropka, C. Frank-Rotsch, F. M. Kießling; *A decade of* KRISTMAG® *technology in crystal growth*; 5th German-Swiss Conference on Crystal Growth; Freiburg, Germany; March 08-10, 2017

R. Zwierz, N. Dropka, A. Glacki, U. Juda, C. Frank-Rotsch; *Flattening of solid-liquid interface in VGF-GaAs growth by various travelling magnetic fields*; XVIII International UIE-Congress on Electrotechnologies for Material Processing (UIE-2017); Hannover, Germany; June 6-9, 2017

S. Kayser, A. Lüdge and K. Böttcher; *Modelling of Lateral-Photovoltage-Scanning-Method (LPS*); 5th German-Swiss Conference on Crystal Growth, Freiburg, Germany, March 08-10, 2017

L. von Helden, M. Schmidbauer, C. Feldt, D. Braun, M. Hanke, Y. Dai, R. Wördenweber, J. Schwarzkopf; *Monoclinic Domains in Strained, Ferroelectric* $K_{0.7}Na_{0.3}NbO_3$ *Thin Films on* $TbScO_3$ *- First Steps to Surface Acoustic Wave Sensors*; International School of Oxide Electronics; Cargese, France; 11-21 April, 2017

D. Braun, L. von Helden, M. Schmidbauer, Y. Dai, R. Wördenweber, J. Schwarzkopf; *Monoclinic Domains in* $K_{0.7}Na_{0.3}NbO_3$ *Thin Films for Surface Acoustic Wave Devices*; European Materials Research Society Fall Meeting; Warsaw, Poland; September 18-21, 2017

C. Ehlers, R. Bansen, D. Uebel, Th. Teubner, T. Boeck; *Growth of Silicon on Reorganized Porous Silicon Substrates by Steady-State Solution Growth for Photovoltaic Applications*; 33rd European Photovoltaic Solar Energy Conference and Exhibition (EU PVSEC 2017); Amsterdam, The Netherlands; September 25-29, 2017

F. Lange, R. Bansen, Th. Teubner, T. Boeck; *Growth of Si, Ge, and SiGe Nanowires*; Austrian MBE Workshop 2017; Wien, Austria; September 28-29, 2017

D. Braun, L. von Helden, M. Schmidbauer, Y. Dai, R. Wördenweber, J. Schwarzkopf; *Strain engineering in* $K_xNa_{1-x}NbO_3$ *epitaxial films: a pathway for different piezoelectric applications*; EMRS Fall Meeting in Warsaw, Poland,September 18–21, 2017

L. Bogula, T. Markurt, J. Schwarzkopf; *Homoepitaxial growth of* $SrTiO_3$ *thin films by pulsed laser deposition*; Gra-FOX annual Meeting, July 20-21, 2017

K. Dadzis, R. Menzel, H. Riemann, N. V. Abrosimov; *Analysis of the melting process of silicon granulate*; 5th German-Swiss Conference on Crystal Growth; Freiburg, Germany, March 08-10, 2017

K. Dadzis, R. Menzel, H. Riemann, N. V. Abrosimov; *Thermal simulation of silicon crystal growth using a granulate crucible*; 7th International Workshop on Crystal Growth Technology; Potsdam, Germany; July 2-6, 2017

R. Zwierz, D. Siche, K. Kachel, S. Golka, M. Bickermann, N. Jankowski, A. Hoffmann; *Vapour phase epitaxy of thick GaN:C layers using HCN precursor gas*; E-MRS Fall Meeting, Warsaw, Poland, September 19-22, 2017

S. Kayser, A. Lüdge, K. Böttcher; *Computational simulation of an LPS (Lateral Photovoltage Scanning) measurement setup*; 5th German-Swiss Conference on Crystal Growth, Freiburg, Germany, March 08-10, 2017

S. Kayser, A. Lüdge, K. Böttcher; *Computational simulations of the Lateral-Photovoltage-Scanning-method*; VIII International Scientific Colloquium: Modelling for Materials processing; Riga, Latvia;
September 21 – 22, 2017

R. Bansen, C. Ehlers, D. Uebel, Th. Teubner, T. Boeck; *Monocrystalline thin-film absorbers by steady-state solution growth*; 27th Photovoltaic Science and Engineering Conference (PVSEC-27); Otsu, Japan; November 2017

T. Markurt, M. Niu, C. Guguschev, D. Kok, K. Irmscher, M. Kociak, and M. Albrecht; *Investigation of point defect diffusion and clustering processes in* $SrTiO_3$ *bulk crystals*; 20th Microscopy of Semiconducting Materials Conference; Oxford, UK; April 9 – 13 April, 2017

Appendix: **Patents**

Granted

S. Ganschow, R. Bertram, D. Klimm, P. Reiche, R. Uecker
Verfahren und Anordnung zur Herstellung von ZnO-Einkristallen
DE 10 2004 003 596.2

Ch. Frank-Rotsch, P. Rudolph, R.-P. Lange, O. Klein, B. Nacke
Vorrichtung und Verfahren zur Herstellung von Kristallen aus elektrisch leitenden Schmelzen
DE 10 2007 028 548.7
08784553.3 (DK, ES, FR, NO)
KRIST*MAG*®

R.-P. Lange, M. Ziem, D. Jockel, P. Rudolph, F. Kießling, Ch. Frank-Rotsch, M. Czupalla, B. Nacke, H. Kasjanow
Vorrichtung zur Herstellung von Kristallen aus elektrisch leitenden Schmelzen
DE 10 2007 028 547.9
08784554.1 (DK, ES, FR, NO)
KRIST*MAG*®

Ch. Frank-Rotsch, P. Rudolph, R.-P. Lange, D. Jockel
Vorrichtung und Verfahren zur Herstellung von Kristallen aus elektrisch leitenden Schmelzen
DE 10 2007 046 409.8
KRIST*MAG*®

P. Rudolph, M. Ziem, R.-P. Lange
Vorrichtung zum Züchten von Einkristallen aus elektrisch leitfähigen Schmelzen
DE 10 2007 020 239.5
KRIST*MAG*®

R. Fornari, S. Ganschow, D. Klimm, M. Neubert, Schulz
Verfahren und Vorrichtung zur Herstellung von Zinkoxid-Einkristallen aus einer Schmelze
DE 10 2007 006 731.5

P. Rudolph, M. Ziem, R.-P. Lange, D. Jockel
Vorrichtung zur Herstellung von Kristallen aus elektrisch leitenden Schmelzen
DE 10 2008 035 439.2

F. Büllesfeld, U. Sahr, W. Miller, P. Rudolph, U. Rehse, N. Dropka
Verfahren zum Erstarren einer Nichtmetall-Schmelze
DE 10 2008 059 521.7
09 749 132.8 (DK, ES, IT, NO, R, GB)

R. Fornari
Vorrichtung und Verfahren zur Züchtung von III-Nitrid-Volumenkristallen
08 161 254.1 (DE, PL, FR, GB, SE)

P. Rudolph, R.-P. Lange, M. Ziem
Vorrichtung zur Herstellung von Sililziumblöcken
DE 10 2009 045 680.5

N. Dropka, P. Rudolph, U. Rehse
Verfahren und Anordnung zur Herstelllung von Kristallblöcken von hoher Reinheit und dazugehörige Kristallisationsanlage
DE 10 2010 028 173.5

H. Riemann, N. Abrosimov, J. Fischer, M. Renner
Verfahren und Vorrichtung zur Herstellung von Einkristallen aus Halbleitermaterial
EP 2 504 470 (NO, ES, NL, FR, DK, GB, BE, IT)

N. Dropka, Ch. Frank-Rotsch, M. Ziem, P. Lange
Verfahren und Vorrichtung zur gerichteten Kristallisation von Kristallen aus elektrisch leitenden Schmelzen
DE 10 2012 204 313.6

N. Dropka, Ch. Frank-Rotsch, P. Rudolph, R.-P. Lange, U. Rehse
Kristallisationsanlage und Kristallisationsverfahren zur Herstellung eines Blocks aus einem Material, dessen Schmelze elektrisch leitend ist
DE 10 2010 041 061.6

O. Klein, F. Kießling, M. Czupalla, P. Rudolph, R.-P. Lange, B. Lux, W. Miller, M. Ziem, F. Kirscht
Verfahren und Vorrichtung zur Züchtung von Kristallen aus elektrisch leitenden Schmelzen, die in der Diamant- oder Zinkblendestruktur kristallisieren
DE 10 2009 027 436.7

H. Riemann, N. Abrosimov, J. Fischer, M. Renner
Verfahren und Vorrichtung zur Herstellung von Einkristallen aus Halbleitermaterial
DE 10 2010 052 522.7
10801372.3 (EP), 13/511,751 (US), 2012-540285 (JP)

F. Kießling, Ch. Frank-Rotsch, N. Dropka, P. Rudolph
Verfahren zur gerichteten Kristallisation von Ingots
DE 10 2011 076 860.2

Z. Galazka, R. Uecker, R. Fornari
Method and apparatus for growing indium oxide (In2O3) single crystals and indium oxide (In2O3) single crystal
EP2841630B1 (DE, BE, FR, GB, IT), JP2018501184

Appendix: **Patents**

Pending

U. Rehse, P. Rudolph, W. Miller, N. Dropka,
F. Büllesfeld, U. Sahr
Method for the solidification of a non-metal melt
WO002012060802A3 (CN, US, TW)

R. Fornari, F. Kießling, P. Rudolph, V. Trautmann
Kristallisationsanlage und Kristallisationsverfahren
DE 10 2009 046 845.5

T. Boeck, R. Fornari, R. Heimburger, G. Schadow,
J. Schmidtbauer, H.-P. Schramm, T. Teubner
Kristallisationsverfahren zur Erzeugung kristalliner Halbleiterschichten
DE 10 2010 044 014.0

M. Wünscher, H. Riemann
Vorrichtung für das tiegelfreie Zonenziehen von Kristallstäben
DE 10 2012 022 958.8
PCT/DE2013/000627

N. Dropka, Ch. Frank-Rotsch, P. Lange, P. Krause
Kristallisationsanlage und Kristallisationsverfahren zur Kristallisation aus elektrisch leitenden Schmelzen sowie über das Verfahren erhältliche Ingots
DE 10 2013 211 769.8
PCT/EP2014/059684

A. Dittmar, C. Hartmann, J. Wollweber, U. Degenhardt,
F. Stegner
Keimhalter einer Einkristallzüchtungsvorrichtung, Einkristallzüchtungsvorrichtung und Kompositwerkstoff
DE 10 2014 017 021.7

Z. Galazka, R. Uecker, D. Klimm, M. Bickermann
Method for growing beta phase of gallium oxide (ß-Ga_2O_3) single crystals from the melt contained within a metal crucible
EP 15150582.3, PCT/EP2015/079938

A. Dittmar, C. Hartmann, J. Wollweber, M. Bickermann
(Sc, Y): Einkristalle für Gitter-angepasste AlGaN Systeme
DE 10 2015 116 068.4, PCT/EP2016/070539

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Appendix: Teaching and Education

Prof. Dr. Matthias Bickermann

- *Kristallzüchtung II: Methoden und Anwendungen;* Technische Universität Berlin, Institut für Chemie, WS 2016/17, WS 2017/2018
- *Kristallzüchtung I: Grundlagen und Methoden;* Technische Universität Berlin, Institut für Chemie; SS 2017
- Forschungspraktikum: Betreuung vom Studienenden am IKZ; Technische Universität Berlin, Institut für Chemie

PD Dr. habil. Detlef Klimm

- *Phasendiagramme;* Humboldt-Universität zu Berlin, Institut für Chemie, WS 2016/17, 2017/18
- Versuch *„Phasendiagramme"* im Fortgeschrittenen-Praktikum der Physik; Humboldt-Universität zu Berlin; WS 2016/17, SS 2017, WS 2017/2018
- Gastvorlesungen an der Humboldt-Universität zu Berlin (Festkörperchemie, Prof. Kemnitz), Technische Universität Berlin (Keramische Werkstoffe, Prof. Gurlo), TU Bergakademie Freiberg (Prof. Heide)

apl. Prof. Dr. Dietmar Siche

- *Kristallzüchtung;* Brandenburgische Technische Universität Cottbus-Senftenberg, Blockveranstaltung; SS 2017

Doctoral theses (ongoing)

Mariia Anikeeva
Transmission Electron Microscopy of Short Period Superlattices for Rational (In,Ga)N

Laura Bogula
MOCVD of perovskite oxide films

Leonardo Cancellara
TEM-Untersuchungen an AlN/AlGaN – Strukturen für LEDs

Elena Castellano-Hernández
Spektroskopische Charakterisierung und Laser-Experimente an Tb^{3+} -dotierten Kristallen

Christian Ehlers
Wachstum und Charakterisierung von Silizium aus Zinn Lösungen für die Photovoltaik

Owen Ernst
Wachstum und Charakterisierung von CIGSe-Absorberinseln für Mikrokonzentrator Solarzellen

Katharina Eylers
Wachstum und Charakterisierung von $Cu(In,Ga)Se_2$ Absorbern für Mikrokonzentratorsolarzellen

Andreas Fiedler
Electrical and Optical Characterization of the Transparent Semiconducting Oxide beta-Ga_2O_3

Ivan Gamov
Point defects and their impact on temperature-dependent properties in AlN bulk crystals

Leonard von Helden
Characterization of Domain Structures in Strained Ferroelectric $Ka_{1-x}Na_xNbO_3$ Thin Films

Felix Lange
Functional Materials and Film Systems for Efficient Energy Conversion (FuSion)

Stefan Kayser
Charakterisierung mono- und multikristalliner Halbleiter wie SiGe, Si, Ge_{1-x} und GaAs mit LPS- und SPL-Methoden

Joanna Moneta
Transmission Electron Microscopy of Short Period Superlattices for Rational (In, Ga)N

Lena Schmack
New Ferroelectric Single Crystals:
A Study of the $LiNb_{1-.x}{\cdot}Ta_x\ O_3$ Solid Solution

Julian Stover
Physics and control of defects in oxide films for adaptive electronics

David Uebel
Wachstum und Charakterisierung von Silizium auf kostengünstigen Subtraten

Nora Wolff
Züchtung von Delafossit-Substratkristallen

Charlotte Wouters
Transmission Electron Microscopy Studies on Phase Formation in Group III-Sesquioxides

Appendix: Teaching and Education

Doctoral theses (completed)

Dorothee Braun
Strain-phase relations in lead-free ferroelectric $K_xNa_{1-x}NbO_3$ epitaxial films for domain engineering

Dirk Kok
Influence of the growth conditions on the optical properties of $SrTiO_3$

Natalia Stolyarchuk
Transmission electron microscopy study of polarity control in III-N films grown on sapphire substrates

Diploma, Master and Bachelor theses (completed)

Owen Ernst, Master
Lokale Strukturbildung von Indium auf amorphen Oberflächen

Dennis Meiling, Master
Evaluation of orientation dependent gallium diffusion during MOVPE growth of gallium oxide

Sascha Schaller, Bachelor
Wachstum von kristallinem Silizium auf Glas durch Züchtung aus metallischer Lösung

David Uebel, Master
Laser-induced Removal of SiO_x from Si Seed Structures for Epitaxial Growth of Absorber Layers for Low Cost Solar Cells

Max Jürgen Wolfgang Schmidt, Master
Einflüsse von Schmelzlösungsmitteln auf die Züchtung von Calciumtitanat-Einkristallen

Professional Education at IKZ (completed)

Sebastian Schermer
Cutting machine operator

Appendix: **Membership in Committees**

Committees

Prof. Dr. M. Bickermann
- IGAFA e.V. - Initiativgemeinschaft Außeruniversitärer Forschungseinrichtungen in Adlershof e.V., member of the board

PD Dr. habil. Detlef Klimm
- Commission on Crystal Growth and Characterization of Materials of International Union of Crystallography (IUCr), consultant

Dr. Wolfram Miller
- Deutsche Gesellschaft für Kristallzüchtung und Kristallwachstum (DGKK), chairman
- European Network of Crystal Growth (ENCG), coordinator

Dr. Christiane Frank-Rotsch
- Deutsche Gesellschaft für Kristallzüchtung und Kristallwachstum (DGKK), secretary
- International Organization for Crystal Growth (IOCG), member of the council
- European Network of Crystal Growth (ENCG), member of the council

Conference committees

Prof. Dr. Matthias Bickermann
- International Workshop on UV Materials and Devices 2017 (IWUMD-2017); Fukuoka, Japan; November 14 – 18, 2017; member of the program committee
- 10th International Workshop on Bulk Nitride Semiconductors (IWBNS-X); Nuuksio, Espoo, Finnland; September 18-22, 2017; member of “International Advisory Committee”
- 7th International Workshop on Crystal Growth Technology (IWCGT-7); Potsdam, Germany; July 2 – 6, 2017; conference organizer

Dr. Christian Kränkel
- Advanced Solid State Lasers Conference (ASSL); Nagoya, Japan; May 01-05, 2017 ; member of the program committee of section „Materials”
- Conference on Lasers and Electro-Optics (CLEO/Europe); Munich, Germany; June 25-29, 2017; member of the program committee of section “CA - Solid State Lasers”
- Europhoton Conference; Barcelona, Spain; September 02-07, 2017; member of the program committee
- Laser Technology and Industrial Laser Conference (SPIE Photonics West); San Francisco, USA; January 28 - February 02, 2017; member of the program committee of the section “Solid State Lasers XXVII: Technology and Devices”

Dr. Wolfram Miller
- 5th German Swiss Conference on Crystal Growth (GSCCG-5/DKT 2017); Freiburg, Germany; March 8 – 10, 2017; member of scientific program committee

Editorial committees

Prof. Dr. Matthias Bickermann
Progress in Crystal Growth and Characterization of Materials, Elsevier, associate editor

Dr. Christian Kränkel
Optics Express, Optical Society of America, associate editor

Dr. Wolfram Miller
Crystals, MDPI, member of editorial board

apl. Prof. Dr. Dietmar Siche
Crystal Research & Technology, Wiley-VCH, member of editorial board

Appendix: Guest Scientists

01.01.2017 – 31.12.2017

Dr. Saud Bin Anooz
01.06. – 30.09.2017
Hadhramout University of Science and Technology,
Physics Department,
Republic of Yemen

Maxim Dzemesh
30.08. – 03.10.2017
Belarussische Nationale Technische Universität Minsk,
Belarus

Prof. Dr. Kozo Fujiwara
22.05. – 30.05.2017
Institut for Materials Research (IMR),
Tohoku University,
Japan

Prof. Dr. Petr G. Sennikov
01.07. – 31.08.2017
RAS – Institute of Applied Physics,
Russia

Sakari Sintonen
01.02.2015 – 31.07.2017
Aalto University,
Department of Micro- and Nanosciences,
Finland

Hiroki Tanaka
07.08. – 31.12.2017
Toei University Tokyo,
Japan

Appendix: Colloquia at the IKZ

Dr. Catherine Dubourdieu
“Epitaxial complex oxides heterostructures”
Institut Funktionale Oxide für die energieeffiziente IT, HZB-Berlin
January 25, 2017

M. Sc. Diana Karsch & M. Sc. Sven Jachalke
“Charakterisierung und Anwendung pyroelektrischer Materialien“
Inst. für exp. Physik der TU BA Freiberg
AG Verbindungshalbleiter und Festkörperspektroskopie
February 3, 2017

Dr.-Ing. Björn Hinze
“Einkristalle in der Luftfahrt“
Materials and Manufacturing Capability Acquisition (ET-M3) Rolls-Royce, Deutschland
February 10, 2017

Dr. Vladimir Roddatis
“Environmental Transmission Electron Microscopy of $Pr_xCa_yMnO_{3+z}$ materials“
Institut für Materialphysik, Universität Göttingen
February 24, 2017

Dr. Joseph Janicsko
“GERDA Phase II, from the concept to the first results”
TU München E 15, Chair for Experimental Physics and Astroparticle Physics
April 07, 2017

Prof. Dr.-Ing. Jostein Grepstad
“Controlling topological spin textures in epitaxial oxide thin film micromagnets“
Norwegian Univ. of Science and Technology (NTNU)
Dept. of Electronics and Telecommunications, Nanoelectronics and Photonics research group
April 21, 2017

MSc Frederik Steib
“Pulsed Sputter Deposition of group-III nitrides“
TU Braunschweig, Institut für Halbleitertechnik
May 05, 2017

Dr. Yuansu Luo
“Thermophysical properties of SiGe-melt measured under microgravity conditions“
I. Physikalisches Institut, Universität Göttingen
June 09, 2017

PD Dr. Dmitri V. Berkov
“Multiscaling dislocation modelling und calculation of dislocation-induced stress“
Research Director, General Numerics Research Lab e.V., Jena
June 16, 2017

Prof. Dr. Zuo-Guang Ye
“Morphotropic Phase Boundary and Structure-Property Relations in High-Performance Piezo-/ferroelectrics“
Simon Fraser University, Burnaby, British Columbia, Canada
July 05, 2017

Dr. Hans Boschker
“A two-dimensional interface superconductor with unconvential gap behaviour and in-gap states“
Max-Planck-Institut für Festkörperforschung,
AG Festkörper-Quantenelektronik
July 17, 2017

Prof. Dr.-Ing. Ulf Schümann
“Die Herausforderungen des Packaging von Wide-Bandgap Halbleitern und deren Einsatz in leistungselektronischen Schaltungen“
Institut für Elektrische Energietechnik, Fachbereich Informatik und Elektrotechnik der FH Kiel – Hochschule für angewandte Wissenschaften
August 18, 2017

Prof. Dr. Liang Wu
“Novel Approach to Spontaneously Grow High-quality Freestanding AlN Single Crystals on Pre-sintered AlN Powder Source by PVT Method“
School of Materials and Science Engineering
Shanghai University, China
August 25, 2017

Prof. Dr. Sven Rogge
“Engineered quantum matter“
School of Physics, ARC Centre for Quantum Computation and Communication Technology, University of New South Wales, Sydney, Australia
October 13, 2017

Prof. Dr. Jürgen Schreuer
“Elasticity - A powerful probe for the exploration of structure/property relationships“
AG Kristallphysik, Institut für Geologie, Mineralogie und Geophysik
Ruhr-Universität Bochum
November 10, 2017

Appendix: **External Funding**

International programs

CHEETAH: Cost-reduction through material optimisation and Higher EnErgy output of solAr pHotovolatics modules - joining Europe's Research and Development efforts in support of its PV industry; EU, 2014-2017

Short Period Superlattices for Rational (In,Ga)N (SPRInG); EU, 2015-2018

Programs of Federal Ministry of Education and Research (BMBF) and Federal Ministry of Economics and Technology (BMWi)

InTerFEL: Zeitaufgelöste und nichtlineare Infrarot- und Terahertz-Spektroskopie mit einem FEL; BMBF, 2014-2017

GERDA: GERmanium Detector Arry zum Nachweis des neutrinolosen doppelten Betazerfalls in 76Ge; BMBF, 2017-2020

Entwicklung von Faraday-Rotatoren mit stark verbesserten Eigenschaften auf der Grundlage von Kalium-Terbium-Fluorid (KTb_3F_{10}) und anderen innovativen Materialien (IsoNova); Teilvorhaben: Technologieentwicklung zur Herstellung von KTb_3F_{10} (KTF)-Kristallen; BMBF, 2017-2020

Entwicklung von graphitfreien keramischen Halbzeugen für die Silizium-Kristallzüchtung (CleanSi); BMWi, 2015-2017

Entwicklung einer Plasmafackel für die Abscheidung von halbleiterreinem AlN zur Herstellung von Sputtertargets (PlasNiTar2.0); BMWi, 2015-2017

Advanced UV for Life – Verbundvorhaben: AlN-Substrate; BMBF, 2015-2017

KrisNet: Entwicklung, Umsetzung und Professionalisierung eines Verwertungskonzepts am Leibniz-Institut für Kristallzüchtung; BMBF, 2015-2018

Verbundprojekt: Galliumoxid Leistungselektronik für hocheffiziente, kompakte Energiekonverter – Oxikon; Entwicklung von halbleitenden n-Typ β-Ga_2O_3 Schichten für Leistungsbauelemente; BMBF, 2017-2020

Erforschung und Qualifizierung innovativer Lasermaterialien und -kristalle; BMBF, 2016-2018

Programs of Länder government

Applikationslabor für Materialien der Oxidelektronik; EFRE, 2017-2020

Leibniz Association

Silizium Granulat Eigentiegelverfahren, 2016-2019

Leibniz ScienceCampus: Growth and Fundamentals of Oxides for Electronic Applications (GraFOx), 2016-2020

Physics and control of defects in oxide films for adaptive electronics, 2017-2019

Barium stannate based heterostructures for electronic applications, 2018-2021

DFG

Entwicklung einer Züchtungstechnologie für semi-isolierende GaN-Substrate und Untersuchung der in-situ Kohlenstoffdotierung, 2014-2017

Lokal gewachsene $Cu(In,Ga)Se_2$-Mikroinseln für Konzentratorsolarzellen; 2015-2018

Züchtung von Delafossit-Substratkristallen, 2016-2019

Kontrolle der Domänen in verspannten, bleifreien Alkaliniobat-Dünnschichten zur Einstellung piezoelektrischer Koeffizienten, 2016-2017

Modellbasierte Steuerung und Regelung des Vertical-Gradient-Freeze-Kristallzüchtungsprozesses mit Hilfe verteiltparametrischer Methoden, 2016-2019

Elektromechanische Eigenschaften und atomarer Transport in AlN-Volumenkristallen bei hohen Temperaturen, 2017-2020

OXITHERM - Ballistischer Wärmetransport in dünnen und ultradünnen Oxidschichten, 2017-2020

Appendix: **External Funding**

Funding by partners from industry and other institutions

Growth and characterization of new oxide crystals for piezoelectric sensors; Kistler Instrumente AG, Winterthur, Switzerland, 2005-2019

KILOGRAMM-3; Physikalisch-Technische Bundesanstalt, Braunschweig, Germany, 2015-2019

Plasmaabscheidung von GaN-Bauelementen PlanB - Elektronenmikroskopische Analyse und Modellierung der Wachstums- und Relaxationsprozesse von PSD InAlGaN-Schichten auf Saphir und Silizium Substraten; Osram Opto Semiconductors GmbH, 2014-2017

Study of growth mechanism and planar defect formation in β-$(Al_xGa_{1-x})_2O_3/Ga_2O_3$ heterostructures grown by MOVPE on differently oriented β-Ga_2O_3 substrates; Air Force Office for Scientific Research (AFOSR), USA, 2017-2020

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