BOOK of ABSTRACTS

3rd GERMAN POLISH CONFERENCE ON CRYSTAL GROWTH

GPCCG3

17 – 21 March 2019, Poznan, Poland

ENCG
European Network of Crystal Growth

3rd German Polish Conference on Crystal Growth (GPCCG-3)

Poznań, 17-21 March 2019

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3rd German Polish Conference on Crystal Growth (GPCCG-3)

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Dear Participants,

It is our great pleasure to welcome you to Poznań for the third edition of a joint German-Polish Conference on Crystal Growth. GPCCG-3 is an international Conference under the auspices of the Polish Society for Crystal Growth (PSCG) and the German Association of Crystal Growth (DGKK).

In 2006 the first joint meeting was held in Berlin followed by the conference in Frankfurt (Oder) and Słubice five years later. During that time photovoltaic was at full swing in Europe and materials for photovoltaics, mostly silicon-based ones were one of the key topics in 2011. At that point things have changed completely. We are very happy that at GPCCG-3 there are many contributions on novel materials with possible great potential in electronics and photonics. On the other side there also many contributions for classical materials like silicon and GaN.

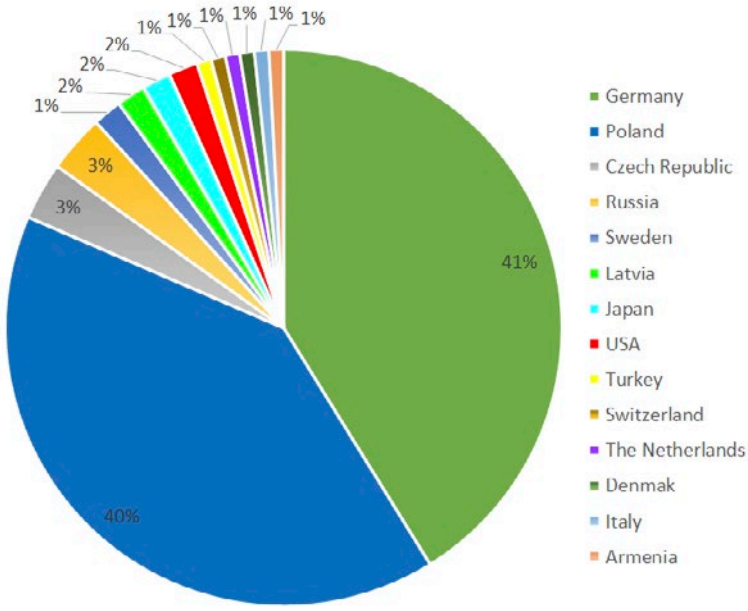
The programme reflects the variety and high standard of research in both countries. It is also a great pleasure to welcome the contributors and participants from other countries all over the world. We hope you all will enjoy the scientific programme, the discussions, and the atmosphere in general.

The conference is hosted by the Poznan University of Technology which celebrates its 100^{th} anniversary this year. Founded in 1919 as the Higher School of Machinery it became the Poznan University of Technology.

We wish all of you an enjoyable stay in Poznań.

Dobrosława Kaprowicz & Wolfram Miller
Chairmen of GPCCG-3

GPCCG-3 Participants coming
from different countries



The German Association for Crystal Growth (DGKK) and the Polish Society for Crystal Growth (PTWK) promote research, education, and technology in the areas of crystal growth, epitaxy, and nano crystalline systems.

Single crystals and epitaxial layers of high quality are the basis of today life. Equipment in information & communication technology, solid-state lighting, energy technology, mobility, and others consists in its relevant parts of devices based on crystals.

Because of their high structural perfection crystals allow an optimal exploitation of the material functionality. There is an ongoing development of new materials with improved and new properties to provide better performance and to reduce energy consumption.

Silicon crystal grown by the Czochralski method. Diameter is 200 mm.
(Photo: Siltronic AG, Burghausen)

Bulk crystals are mainly grown from melt. This is the fastest way to obtain large single crystals of high quality and by this also the cheapest (e.g. silicon crystals for electronic devices). In certain cases this is not possible and crystals are grown from solution (e.g. CaF_2 for lenses in lithography) or gas phase (e.g. silicon carbide for power electronics).

Bulk crystals can be used as pieces, e. g. for lasers, or are cut into wafers. Wafers are the substrates for epitaxial growth of functional layers and structuring by lithography to obtain chips for computers, smart phones etc. .

The most prominent method for growth from melt is the Czochalski method. It is named after the Polish scientist (1885-1953), who invented this method in 1916 when he was working at Allgemeine Elektricitäts-Gesellschaft (AEG) in Oberschöneweide, at that time a suburb of Berlin.

DGKK was founded in 1970.
Today it has about 400 members including companies and research institutes.
Further information: https://www.dgkk.de

PTWK was founded in 1991.
Today it has about 200 members.
Further information: http://www.ptwk.org.pl/

DGKK and PTWK are members of the International Organization for Crystal Growth (IOCG) and the European Network of Crystal Growth (ENCG).

2nd German Polish Conference on Crystal Growth (GPCCG 2011) in Frankfurt (Oder) and Słubice
(Photo: Thomas Jauß, Freiburg)

There is a long tradition in cooperation of DGKK and PTWK. After joined conferences in 2006 and 2011 we will have the 3rd German Polish Conference on Crystal Growth (GPCCG 2019) in 2019 to be held in Poznan.

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Phase separations and nematicity in dilute magnetic materials

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Semiconductors [1] and topological materials [2] doped with magnetic impurities attract a considerable attention due to a fascinating physics and nonospintronic functionalities associated with exchange coupling between band carries and localized spins. However, there is a growing amount of evidences that *d*-shells of magnetic impurities contribute also to bonding, which can affect their spatial distribution and modify key properties, such as magnetic ordering temperature [3]. It has recently been experimentally demonstrated that the resulting phase separation (spinodal decomposition) can be anisotropic and result in the hitherto puzzling rotational symmetry breaking (i.e. nematic characteristics) revealed in a certain class of dilute magnetic semiconductors [4]. This finding put in a new light a possible origin of nematicity in other systems, such as unconventional superconductors and modulation doped semiconductor quantum wells, in which rotational symmetry breaking has so far been assigned to unidirectional spontaneous ordering of spin, orbital or charge degrees of freedom.

PLENARY

References

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PL.2

PLENARY

Exploring 1D semiconductor crystal growth at the nanoscale using in-situ TEM

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Semiconductor nanowires have emerged as a highly promising technology for next-generation electronics and photonics, in particular due to their potential for forming novel metastable crystal phases, complex alloys and heterostructure combinations not achievable in bulk semiconductors. The most common method of fabricating these structures is the vapor-liquid-solid mechanism, which makes use of a catalytic liquid metal droplet. The development of these materials requires a fundamental understanding of how they form. Since nanowire growth is performed in a vapor phase atmosphere at high temperature, the dynamic processes controlling their formation cannot be directly deduced by analyzing only the final grown nanostructure. As such, the current mechanistic understanding of the synthesis process is insufficient for achieving the promised level of control.

In order to address this challenge, we use *in-situ* TEM imaging combined with *in-situ* compositional analysis to study the processes occurring at the interface between the metal droplet and nanowire, and how these control the final structure. Nanowires are grown in a Hitachi HF3300S aberration-corrected environmental TEM connected to a chemical vapor deposition system designed for III-V semiconductor growth. Growth is performed on a SiNx-based MEMS heating chip mounted on a holder with two separate microtubes for supplying the precursor gases. High resolution, high-frame-rate videos enable us to determine the rate at which individual semiconductor bilayers form, along with the interface morphology, catalyst geometry and nanowire crystal structure. The elemental composition of the catalyst is measured by energy dispersive X-ray spectroscopy as a function of the growth parameters. We identify different ‘regimes’ in which growth occurs that can be identified by the composition and structure of the catalyst, and determine the relationship between the accessible experimental parameters and the steps in the growth mechanism.

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PL.3

Solid state single crystal growth of three-dimensional faceted LaOFeAs crystals

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Soon after the discovery of iron-based superconductors, single crystals of various pnictide families were successfully grown using self-flux growth. However, LaOFeAs single crystals are hard to be obtained via flux growth[1,2] although crystals could be successfully synthesized using high-pressure-high-temperature synthesis[3] but this method is limited to rather small crystals and also did not lead to a reasonable growth along the *c* direction, so far. This lack of large, well-faceted and 3-dimensional crystals is currently hindering their detailed investigation.

Solid state single crystal growth (SSCG) is a crystal growth technique where crystals are grown from a polycrystalline matrix. Here, we present single crystals of the iron pnictide LaOFeAs grown via SSCG using NaAs as a liquid phase to aid crystallization. The size of the as-grown crystals are up to $2 \times 3 \times 0.4\,mm^3$. Typical for this method, but very uncommon for crystals of the pnictide superconductors and especially for the oxypnictides, the crystals show pronounced facets and considerable growth in *c* direction[4]. Current research aims to further understand details of the growth mechanism and to explore growth of crystals with different types of doping using the SSCG.

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PLENARY

PL.3

PLENARY

3D Metamaterials: Rationally Designed Artificial Crystals

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Metamaterials are rationally designed three-dimensional composites made of one or more bulk ingredient materials that allow for effective-medium properties going beyond ("meta") those of the ingredient materials, qualitatively and/or quantitatively [1].

In this talk, I focus on three-dimensional (3D) crystalline metamaterials. After an introduction into the concept, I discuss recent examples. This includes artificial cubic chainmail-like crystals leading to a sign reversal of the effective isotropic Hall coefficient, anisotropic crystals exhibiting the parallel Hall effect, chiral cubic crystals showing twist effects forbidden in Cauchy elasticity as well as the phenomenon of "mechanical activity", and cubic crystals exhibiting a sign reversal of the isotropic thermal expansion coefficient and a negative static effective compressibility, respectively.

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Session talks

1A.1

Bulk growth of GaN – status, perspectives and trends

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INVITED

GaN-based devices, like light emitting diodes (LEDs) or high electron mobility transistors (HEMTs), are mainly built on foreign substrates (sapphire, silicon, or silicon carbide). Nevertheless, the nitride community is still looking for GaN substrates in order to advance the existing generation and develop the new one of the products. For many years work on developing bulk GaN has mainly been motivated by the laser diodes market. Today, the main driving force for crystallizing bulk GaN seems to be the electronic industry and a demand for vertical high power transistors and diodes. For this application GaN wafers of high structural quality and with a high free carrier concentration are necessary. However, crystallization of GaN is quite a challenging process. The gallium nitride compound melts at extremely high temperature (>2200°C) and the nitrogen pressure necessary for congruent melting of GaN is expected to be higher than 6 GPa[1]. Thus, today, it is impossible to crystallize GaN from the melt. This compound should be grown by other techniques requiring lower pressure and temperature. Three main technologies are applied for GaN crystal growth: sodium flux, ammonothermal and Halide Vapor Phase Epitaxy. The first two of them represent growth from solution, the third one growth from the vapor phase. In this paper a current status of these three methods will be described in detail. The main advantages and disadvantages of each method will be presented. A possibility to grow boule of GaN will be analyzed.

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1A.2

ORAL

Defect analysis of ammonothermal GaN and HVPE-GaN grown on ammonothermal GaN seeds

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The ammonothermal growth process allows the realization of GaN crystals with low threading dislocation density (TDD) and flat crystallographic planes. TDDs as low as $5 \cdot 10^3$ cm^{-2} can be achieved for ammonothermal GaN crystals (Am-GaN) which is a 3-4 orders of magnitude lower TDD than hydride vapor-phase epitaxy GaN (HVPE-GaN) crystals grown on a foreign substrate. On the other hand, the ammonothermal growth method implies technological challenges such as the high incorporation of impurities or the elaborate multiplication of the crystals.

One way to overcome these challenges is to use Am-GaN crystals of very high crystallographic quality as seeds for HVPE growth. This allows crystallization of HVPE-GaN crystals of equally high structural quality as Am-GaN with flat crystallographic planes and high purity.

In this work we present a detailed analysis of defect structures of GaN (0001) crystals grown by the ammonothermal method and HVPE crystals grown on ammonothermal GaN seeds. Both, semi-insulating GaN substrates for lateral HEMT devices as well as n-type GaN substrates for vertical Schottky diodes were investigated. Different X-ray topography techniques (laboratory Lang X-ray topography and synchrotron white-beam X-ray topography (SWXRT)) were used to identify extended defects such as TDDs, growth sectors, grain boundaries, etc., with a large field of view within the GaN crystals. Additional, synchrotron X-ray monochromatic rocking curve imaging (RCI) was employed. RCI enables the local determination of the effective misorientation, which results from lattice parameter variation and the local lattice tilt, and the local Bragg position. Maps of the integrated intensity, the FWHM and the peak position angle of the diffracted intensity, calculated from the ensemble of rocking curve images, provide quantitative values related to the local distortion of the crystal lattice. With the combination of the results from the different X-ray diffraction techniques, models for the diverse defect structures of the GaN crystals grown by ammonothermal method and HVPE on ammonothermal GaN are discussed.

1A.3

The growth and properties of mixed $Cd_{1-x}Zn_xTe$ alloys

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Investigated in this work mixed $Cd_{1-x}Zn_xTe$ compounds are interested materials for modern electronics and materials science. CdTe based crystals are promising materials as x-ray and gamma-ray detectors [1] but also substrate for infrared sensors (HgCdTe) [2]. The variation in composition allows tuning of their fundamental parameters like energy band-gap and lattice constant, what is very important from application point of view.

$Cd_{1-x}Zn_xTe$ mixed crystals investigated in this work were grown from the melt by vertical Bridgman-Stockbarger method in the whole range of composition $0<x<1$ that is from one binary crystal (CdTe) to another (ZnTe). The real composition of grown crystals was measured with SEM/EDS method along growth axis. The segregation coefficient of Zn in CdTe matrix has been evaluated as close to unity. The energy gap as a function of the composition was determined from transmission spectroscopy. Thanks to that bowing parameter of this ternary alloy was found to be 0.458. In this work for the first time the systematical study of thermal properties of $Cd_{1-x}Zn_xTe$ alloys from one binary crystal (CdTe) to another (ZnTe) grown by vertical Bridgman technique were undertaken. The thermal diffusivity and effusivity of investigated crystals were derived from the experimental data and allowed calculating the thermal conductivity. Diagrams of the thermal conductivity versus composition were analyzed applying model for mixed semiconducting crystals given by Sadao Adachi [3]. Thanks to that contribution of the thermal resistivity arising from the lattice disorder to the total resistivity of the crystal has been determined.

ORAL

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1A.4

ORAL

Investigation of facet growth in heavily doped silicon single crystals grown by the floating zone technique

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It has been shown that a link between the stability of facet growth, the generation of crystal defects, and constitutional supercooling exists for growing silicon single crystals. In order to obtain a deeper understanding of this relationship, the facet growth and the occurrence of constitutional supercooling were systematically investigated.

For that purpose, heavily As-doped, <100>- and <111>- oriented silicon crystals with 8 mm in diameter were grown by the floating zone technique in a double ellipsoidal mirror furnace. The grown crystals were characterized with regard to the facet lengths, the width of the growth ridge, the shape of the phase boundary, and the emergence of constitutional supercooling.

The results show, that unlike to crystals with large diameter, the edge facet length is not limited to the region of the growth ridge. Based on the observation of constitutional supercooling and temperature gradients determined by the analysis of central facet length and the shape of the phase boundary, a critical v/G ratio was calculated. These parameters were tested to purposefully evoke constitutional supercooling and were compared to the Tiller criterion.

2A.1

Growing High-Volume Synthetic Laser and Faraday Crystals for Medical, Industrial, Scientific and Defense applications – Opportunities in the Present and Future Global Market

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INVITED

Northrop Grumman SYNOPTICS entered into the synthetic laser material business in 1965 and has become an industry leader in the manufacture of solid-state laser materials. Our focus on growth markets in industrial and medical/aesthetic applications was key to the development of a successful crystal growth business. Our strong competitive position is based on repeatable and lean processes to produce large and high-quality boules. Important is also our vertical integration with crystal laser rod, slab and disk fabrication, polishing and coating, as well as facility, supply chain and customer management. SYNOPTICS' one hundred Czochralski growth stations support today's volume products aluminum garnets (RE:YAG and others), gallium garnets (TGG, YSGG, GGG), fluorides (RE:YLF), Alexandrite, Ruby as well as vanadates and perovskites.

SYNOPTICS' R&D team currently develops volume growth techniques for emerging products, for example, RE:LuAG & RE:Lu_2O_3 for scientific and industrial application and Ti:Sapphire for ultra-short pulse lasers and amplifiers. Another new crystal in high demand is KTF, a low-absorption Faraday crystal for high power laser isolators[1]. Other crystals grown by Bridgman or hydrothermal crystal growth technique for mid-IR laser, non-linear optical, scintillator or semiconductor device applications are in various exploratory stages.

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2A.2

ORAL

Flux single crystal growth of nonlinear optical quaternary oxides containing tellurium ions

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Complex tellurium compounds have been extensively investigated in recent years due to their potential applications in nonlinear optics.[1,2] There are two groups of tellurium containing compounds of great interest:

1. Tungstates and molybdates containing Te^{4+} ions. In such compounds d^0 transition metal cations (Mo^{6+} or W^{6+}) and cations with non-bonding electron pairs (Te^{4+}) make them susceptible to the second-order Jahn–Teller effect, and in the consequence cause strong distortion influencing their nonlinear optical (NLO) properties. Good example of such compound is $BaTeMo_2O_9$.
2. Compounds containing Te^{6+} ions also reveal Jahn-Teller effect due to presence of TeO_6 octahedra. Such compounds can contain other components increasing their NLO properties (BO_3 planar groups and cations with stereochemically active lone pair of electrons as in Bi^{3+}). An excellent example of such compound is Bi_3TeBO_9 showing exceptionally strong NLO properties. Moreover Bi^{3+} sites can be effectively substituted with RE^{3+} ions what can lead to self-frequency doubling (SFD) laser applications.

The mentioned above compounds melt incongruently, therefore high temperature solution growth was used to obtain their single crystals. Conditions of crystallization, doping, and limitations encountered during crystallization of $BaTeMo_2O_9$ and Bi_3TeBO_9 will be discussed in details.

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2A.3

Estimation of Sc^{3+} Solubility in Dodecahedral and Octahedral Sites in YSAG:Yb. Influence of the precursor powder morphology and forming conditions on the high optical transmittance of YAG:Yb ceramics

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ORAL

A method for synthesizing YSAG:Yb optical ceramics has been developed. Additionally, the phase composition and unit cell parameters for YSAG, YbSAG, and their solid solutions have been determined. The limits of scandium solubility in the dodecahedral and octahedral sites of garnet crystal lattice have been estimated after annealing at 1600°C. The scandium solubility limit in the dodecahedral sites of YSAG:Yb was found to be 66 ± 2 at.%.[1] The limit of scandium solubility in octahedral sites depended upon the type of garnet-forming cations. The solubility limit for Sc^{3+} decreased from 97.5 ± 0.5 at.% to 68.5 ± 1 at.% with simultaneous increase of ytterbium content in the YSAG-YbSAG system. Therefore, the region of solid solutions existing with a garnet structure in the Al_2O_3-Sc_2O_3-Yb_2O_3-Y_2O_3 system was determined. Samples having scandium in both dodecahedral and octahedral sites, which had a Sc content of ~30 at.%, were synthesized as a precursor for optical ceramic production.

The influence of the dispersity and morphology of ceramic powders on the characteristics of green bodies and the optical transmittance of YAG:Yb (20 at.%) ceramics were considered. The effect of the specific surface area on the relative density of compacts was studied. Increasing in the specific surface area from 1.45 to 12.38 m^2/g was led to decreasing in the relative density of green body compacts from 52 to 38% under the fixed uniaxial pressure of 50 MPa. Increasing of uniaxial pressing value up to 150 MPa was provided the maximum increasing of optical transmission of ceramics. However, increasing in the specific surface area and uniaxial pressure could led to the appearance of macrodefects in ceramic samples. Cold isostatic pressing at 200 MPa after uniaxial pressing at 50 MPa was resulted in increasing of optical transparency and eliminate of macrodefects formation in ceramics. Dispersity and

3A.1

INVITED

Modelling ridge and facet growth in silicon crystals

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Si crystals grown in the <100> orientation have 4 ridges at the crystal surface in the <110> directions where the outer {111} facets are present. The ridge formation is caused by the dependence of undercooling on the interface orientation [1]. The shape and size of the ridge depends on the growth conditions and it can be present in the form of a protrusion or contraction on the surface of the crystal. The ridges can unfavorably influence the monocrystalline growth e.g. increase the thermal stress in the crystal [2].

A 2D model [3] describes the width and the radial and vertical height of the ridge depending on the growth parameters. These dimensions are strongly dependent on the temperature gradient and Voronkov angle, but also parameters such as the crystal diameter, pull rate and interface angle have an impact on the ridge size. However, the model includes considerable simplifications, such as assuming a parabolic ridge shape and a linear dependence of the undercooling as a function of the ridge size.

Therefore a 3D model has been developed, which explicitly calculates the deformed shapes of the melt surface and the crystallization interface. The temperature field inside the crystal and melt is simulated based on the *OpenFOAM* solver. The local crystal growth rate is modelled as a function of undercooling and surface orientation as described in [4]. A quasi-stationary solution is reached when the shape of the triple point line ensures the growth angle necessary for vertical growth at all points.

The results of the 3D model provide the shape of the facet including its prolongation inside the crystal as well as radial, azimuthal and vertical protrusion of the ridge on the crystal surface. The perturbation of the temperature and the shape of the undercooled melt volume at the {111} facet are calculated. The effect of several process parameters such as the crystal diameter and the hot zone design is analyzed on the basis of the floating zone growth process. The ridge dimensions are compared to the 2D model and experimental values for the floating zone grown crystals.

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Silicon crystal growth using high frequency induction heating

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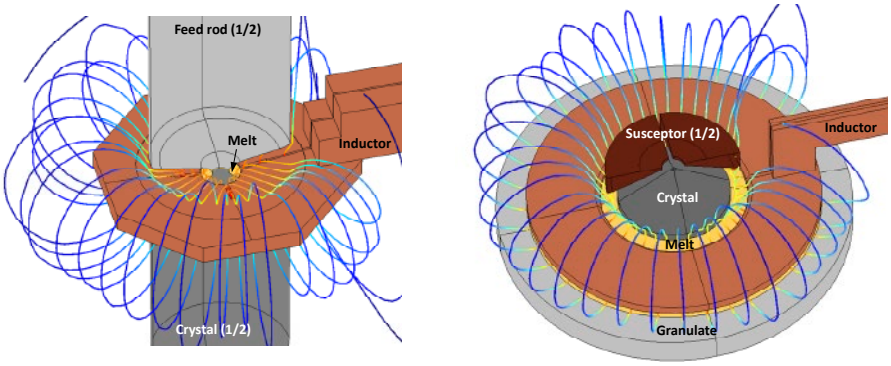
High frequency (HF) induction is the key feature of the well-established floating zone (FZ) growth method for large silicon single crystals[1]. The magnetic field generated by a flat single-turn inductor determines the melting process of a polycrystalline feed rod above the inductor as well as the heat and force balance in a molten zone below. In the recently introduced silicon crystal growth from a granulate crucible (Si-GC) similar inductors are applied to melt silicon granulate as well as to provide suitable temperature distribution in the melt and in the growing crystal including the effect of a self-stabilizing crystal diameter[2].

To a large extent, the development of the FZ and Si-GC methods deals with the design of the HF inductors and related process geometry (e.g., susceptors), see figure below. Numerical modeling of the magnetic and thermal fields has been employed in the literature[1], however, many important aspects in these models are not well understood. This includes both material models (e.g. liquid films on silicon surfaces) and settings for electrical parameters (inductor current, voltage, and frequency)[3].

The present study will address these open questions from a complementary experimental and numerical perspective. Measurements of electromagnetic and thermal fields will be used to validate the numerical models and to develop a better understanding of HF induction in the FZ and Si-GC processes.



Geometries of FZ (left) and Si-GC (right) methods with calculated magnetic field lines.

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3A.3

ORAL

Modelling of CZ Si crystal shape and point defect distribution using different pull rates and heater powers

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During the growth of silicion (Si) single crystals using the Czochralski (CZ) technique, the crystal pull rate and the heater power are adjusted to maintain a predefined crystal shape. The CZ process is strongly non-stationary, i.e., the temperature and point defect concentration fields depend on their past values.

An important crystal quality criterion is the distribution of the point defects – self-interstitials and vacancies. In experiments by Abe et al. interstitial-rich regions in the crystal were formed by temporarily reducing the crystal pull rate[1]. Experimental results were reproduced by the numerical simulations[2], but the heater power and crystal radius changes were not considered in the model.

In the present study the influence of the pull rate drop and heater power on the crystal shape and point defect distribution is analyzed using the program CZ-Trans[3,4], including the impact of the thermal stresses on the point defects. Crystals with a diameter of 50, 100 and 200 mm are considered, see an example in Fig. 1. To stabilize the crystal radius, a heater power jump is applied. Besides the experimental[1] pull rate drop several other values are also modelled in combination with different heater power jumps.

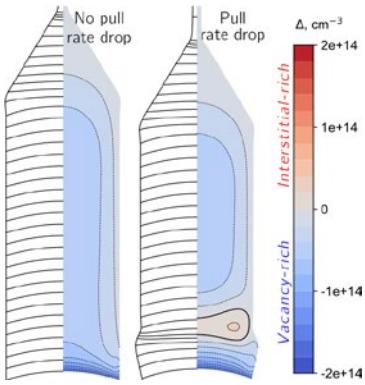

Fig. 1. Crystallization interface shapes plotted each 10 min (left) and point defect concentration difference (right) for 100 mm crystal.

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3A.4

Modeling of the Low Temperature Solution Growth of ZnO Nanorods

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One of the crucial parameters responsible for the growth habit of ZnO nanorods prepared by low temperature solution growth is the level of supersaturation. At different supersaturations, different growth mechanisms[1] and different growth velocities in specific crystallographic directions were observed.[2] By accurately controlling the supersaturation, one can obtain variety of ZnO nanorod morphologies with different physical properties.

In this work, we model the distribution of supersaturation in the nanorod interface using (a) diffusion/reaction model[3,4] and (b) direct modeling of the diffusion equation in COMSOL Multiphysics. Experimentally measured growth velocities and surface coverage ratios of ZnO nanorods grown either on ZnO seed layers or on GaN substrate were used as input data to the models. The results were correlated with the experimentally grown nanorods. The surface morphology of experimentally grown ZnO nanorods were characterized by scanning electron microscopy (SEM), transmission electron microscopy (TEM), and X-ray diffraction (XRD).

ORAL

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4A.1

INVITED

Studies of growth parameters for high purity Ge crystals

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Radiation detectors made of High Purity Germanium (HPGe) are in the front line of fundamental research since their discovery. They dominate the field of the search for neutrinoless double beta decay, nuclear physics and are used for dark matter searches as well.

The growth of HPGe crystals is a non-trivial task. For satisfying the extreme purity requirements a custom built Czochralski furnace is needed and the crystal growth should be carried out in hydrogen atmosphere. Since crystal defects have a detrimental effect on the performance of germanium detectors their number has to be within acceptable limits[1]. To reach the target range for the dislocation density a precise control of the temperature field is needed. In order to build a Cz-furnace that satisfies all the requirements we used numerical modeling assisted design. 3D CFD simulation of the temperature field and 2D axis-symmetric simulations of the growth process were performed. The internal parts of the furnace were designed and built entirely by IKZ.

First results of Czochralski grown 2” Ge single crystals will be presented. The used Ge has been purified in a horizontal multi-zone furnace built by IKZ. The crystals were analyzed by means of Hall-effect measurements and PTIS. EPD measurements were performed to obtain the dislocation density. Some crystals were characterized with the LPS method to gain information on the solid-melt interface during the growth.

For the search for neutrinoless double beta decay HPGe detectors made of germanium enriched in the Ge-76 isotope are needed. To handle the enriched material IKZ sets up the full production chain to produce HPGe crystals as part of the contribution to the LEGEND-200 experiment.

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4A.2

Experimental and theoretical analysis of the growth ridge geometry of Czochralski-grown silicon crystals

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We present a contactless, non-destructive approach based on surface topography to measure the geometrical parameters of the growth ridge and apply it to silicon crystals grown by the Czochralski method. This method gives an easy access to the temperature gradient at the crystal edge during growth.

From theoretical considerations[1], it is well known that the temperature gradient, edge facet growth and the growth ridge are tightly connected. Also the geometry of the growth ridge and its quantitative dependence on the temperature gradient was theoretically predicted[1], but experimental results have not been published yet. Using the results from our experiments for a large variety of industrially grown Czochralski silicon crystals, we confirm the universal validity of that theory[2]. This confirmation in turn allows directly correlating the geometrical parameters of the growth ridge to the temperature gradient. Therefore, fluctuations as well as absolute values of the temperature gradient can be extracted from the measurements, making this a powerful tool for the assessment of crystal growth processes.

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4A.3

ORAL

Buffer development for GaN Power Transistors on 200 mm Silicon Substrates

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GaN-on-Si power devices are gaining more and more attention for applications utilizing an operation voltage of 650V. For this voltage range, a buffer breakdown exceeding 650V together with low buffer dispersion is mandatory. Silicon with diameter up to 200mm has proven to be the substrate of choice for the HEMT due to its cost effectiveness and large diameter availability. Besides the established staircase AlGaN buffer with increasing Al-content we investigate a step-graded AlGaN buffer with a super-lattice (SL) approach. To obtain a buffer layer with a high average Al content AlGaN/AlN seems to be favorable to achieve a high breakdown voltage with reasonable buffer thickness, as electrical breakdown field for AlN is higher than for GaN.

We will present our recent work performed in a G5+ Planetary Reactor® in the 5x200mm configuration by using 725µm thick Si (111) substrates. To enable a high Al fraction in an AlGaN/AlN SL buffer, a new approach is presented. By avoiding relaxation of the buffer, thickness of the structure can be easily engineered. Different growth conditions and modified layer sequences are tested to achieve high breakdown voltages, low buffer dispersion and optimum productivity of the MOCVD tool. This approach will be discussed in detail based on our experimental findings of X-ray analysis, PL mapping and electrical measurements.

Further, the breakdown behavior of a buffer depends on carbon background level in the structure. We have shown, that extrinsic doping of GaN:C by a Hydrocarbon source shows advantages compared to the conventional approach of a low temperature process. We now extent this approach of extrinsic doping to the developed new buffer structures. By this method the carbon level can be well controlled resulting in a uniform buffer breakdown (I_{leak}= 1µA/mm) for a 4.5µm thick structure of about 1000 V across a 200mm wafer. We will report on impact of carbon level on HEMT device properties such as buffer breakdown, carrier mobility and dynamics properties.

5A.1

Synchrotron-based high resolution x-ray diffraction at individual low-dimensional objects using highly focused hard x-rays

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INVITED

Recently focusing optics (as e.g., Fresnel zone plates and refractive lenses), for hard x-ray synchrotron radiation ($\lambda \leq 1$ A) have been developed, which may provide x-ray beam foci of down to 50 nm. A central aim of the discussed studies is the application of nanofocused hard x-rays to high resolution x-ray diffraction, a method which typically uses extended x-ray foci and thus averages over large ensembles. As a consequence measurements have been performed by using setups at ID01 and ID13 of the European Synchrotron Radiation Source (Grenoble), and P06 at PETRAIII (Hamburg) with x-ray beams that were typically between 100 nm and 250 nm in size.

We will briefly introduce the basic experimental idea by a reference experiment, in which individual parts of a single SiGe/Si(001) island (samples were grown by liquid phase epitaxy) have been excited [1], whereas the main part of the contribution will focus on isolated core-shell (In,Ga)N/GaN rods [2].

The incorporation of indium atoms into a GaN matrix offers the possibility to tune the emitted wavelength. This makes the ternary (In,Ga)N alloy embedded into a GaN matrix interesting for a variety of optoelectronic applications.[3] To improve and expand existing group III-nitride based semiconductor technologies researchers have focused their activities on the investigation of low-dimensional structures such as quantum dots or nanowires. For example, core-shell nanowires and rods are discussed as promising candidates for next generation light emitting diodes. If the structures are grown not in an axial but in a core-shell geometry, the optically active area can be significantly increased compared to planar structures. Moreover, concerning GaN the growth of (In,Ga)N quantum wells on non-polar m-planes is especially interesting because the optical performance is not influenced by polarization potentials and the quantum confined Stark effect. Hence, indium content and quantum well width mainly determine the band gap. However, in contrast to planar structures, the structural analysis and the determination of the chemical composition of low-dimensional objects requires new characterization approaches with a focus on spatial resolution, such that fluctuations in the submicrometer regime can be detected.

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5A.2

ORAL

More Insights in Semiconductor Material Quality with Advanced X-ray Topography Imaging

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Characterization of structural defects in semiconductor materials is an important task to understand the influence of the production conditions on the material quality of substrates and epilayers and to correlate the impact of structural defects on the performance and reliability of devices manufactured out of the substrates and epilayers. State of the art characterization of structural defects in single crystalline material like dislocations, dislocation networks, and slip lines is done by defect selective etching (DSE), Cathodoluminescence (CL), Photoluminescence (PL) or by X-ray topography (XRT). Unfortunately DSE is a destructive method and leads to a significant yield loss, especially important for expensive substrate materials. CL could be used only on small samples and needs a high sample preparation effort. CL and PL are applicable only for electrically active defects. PL is not easily available for semiconductors with high band gaps like AlN. XRT measurements were done in the past mostly at synchrotron radiation sources, are highly complex and the accessibility is not satisfying.

Meanwhile advanced X-ray topography tools for use in a laboratory are on the market like the XRTmicron which allows high quality 2D and 3D topograms due to the usage of a novel highly focusing anode. The availability of such advanced tools gives the possibility to investigate crystallographic defects such as the amount and different types of dislocations, slip lines, dislocation networks, (small angle) grain boundaries, inclusions, precipitates, pits, scratches, etc. with high speed and high resolution on full wafer scale on bare wafers, wafers with epilayers, partially processed wafers as well as bonded wafers.

In this contribution we give an overview of the principle of the XRTmicron tool and present examples how it can be used to analyze e. g. the slip line formation in 300 mm silicon wafers, the different kinds of dislocation types (threading screw, threading etch and basal plane dislocations) in 4H-SiC substrates and epilayers, the quality of differentially grown AlGaN layers on sapphire, and the defect propagation in AlN-bulk growth.

5A.3

Surface Enhanced Raman Scattering in a volumetric nanoplasmonic eutectic composite Bi_2O_3-Ag

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ORAL

Plasmonic materials, due to their unique electromagnetic properties, are applied in many areas of research such as photonics, optoelectronics and materials engineering. One of most interesting fields of application is the Surface Enhanced Raman Spectroscopy (SERS), which due to an interaction between plasmons and investigated sample, allows for sensitive measurements of extremely low concentrated solutions or even single molecules.

Current sample preparation methods are based on locally modified surfaces, which are low efficient, time-consuming and expensive. Here we demonstrate a self-organized Bi_2O_3-Ag eutectic material, in which silver forms microprecipitates embedded in bismuth oxide matrix. Post-growth heat treatment of the eutectic composite causes a transformation of as-grown silver phase to metallic nanoparticles, which are responsible for the presence of the Localized Surface Plasmon Resonance (LSPR) with the tunable maximum at ~595 nm.[1]

Performed Raman spectroscopy measurements on LSPR-exhibiting samples has confirmed presence of SERS, which intensity depends on the selected excitation wavelength and corresponds to the extinction coefficient curves. Obtained enhanced Raman spectra of the Bi_2O_3-Ag composite have allowed for phase-structure characterization, including identification of a metastable γ-Bi_2O_3 phase. Corresponding TEM and SAD analysis confirm presence of the γ-Bi_2O_3 structure, which exists in form of 50 nm nanocrystals.[2]

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6A.1

ORAL

In situ observation of interaction between grain boundaries during directional solidification of Si

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The GB distribution in multicrystalline Si (mc-Si) substrates has been confirmed to be an important factor influencing the performance of solar cells. The GB distribution during directional solidification is determined by the growth behavior of GBs at the solid/melt interface and by how GBs interact as they come into contact with each other during growth. An improved understanding of GB growth during solidification would enable a more accurate explanation of the GB distribution observed after solidification.

In this study, the GB interactions were observed in situ during directional solidification. Fig.1 shows the solid/melt interfaces and EBSD mappings for samples A and B, respectively. SAGBs converged with Σ3 GBs at crystal/melt interface and crossed each other without significant change on misorientations. This phenomenon were observed for SAGBs with different rotation axes and misorientations in samples A and B. Since a SAGB is an array of dislocations, the dislocation layers can propagate through Σ3 GBs during solidification. This result implies that those boundary dislocations decomposed into partial dislocations at Σ3 boundary and generated new dislocations at the other side of boundary during solidification. Our observations also showed that SAGBs cannot propagate through Σ9 and Σ27 GBs. The mechanism behind the interactions of SAGBs at Σ3 GBs are different from their interactions at Σ9 and Σ27 GBs.

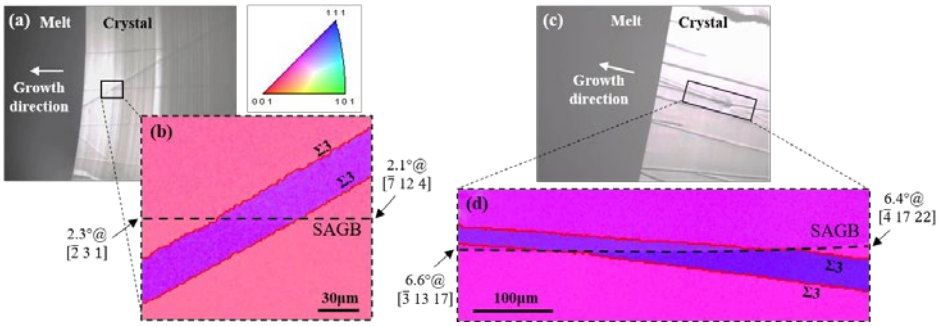

Fig.1 Crystal/melt interface during directional solidification of (a) sample A and (b) orientation mapping of interaction area along growth direction; (c) sample B and (d) orientation mapping of interaction area along growth direction.

6A.2

Adjustment of resistivity for phosphorus doped n-type multicrystalline silicon

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Modern photovoltaic market constantly moves in the direction of solar modules with high efficiency and low cost. Novel solar cell designs are mainly focused on n-type crystalline Si substrate material and can fulfil the requirements of high efficiency. However, due to peculiarities of dopant segregation, industrial n-type phosphorus-doped crystalline ingots show broader resistivity variation in comparison to p-type ones, hence lower yield and higher cost. This phenomenon is observed for both monocrystalline and multicrystalline silicon material (mc-Si). Some specific solutions how to ensure homogeneous resistivity profile of monocrystalline Cz ingots are already available. The approach applicable for directionally solidified (DS) mc-Si is still missing though.

One of the effective ways to assure homogeneous quality of crystallized material is supposed to be crystal growth in a half-open system that implies constant or periodical supply/removal of dopant. In case of DS method, which is limited with sealed growth chamber, the solution lies in effective evaporation of volatile phosphorus dopant throughout the ingot growth.

In this work authors present results for G1-sized (22 x 22 x 12 cm^3) n-type DS mc-Si ingots grown in different conditions (melt mixing, ambient gas flow, ambient pressure etc.) in order to enhance or suppress phosphorus evaporation during growth process. A specific attention is given to enhanced melt stirring by travelling magnetic fields (TMF) since it is an effective method to influence the complex interaction between melt flow, shape and morphology of the solid-liquid interface and impurity transport. It will be shown that the complex approach to melt stirring and gas transport is an effective way to control and tailor resistivity distribution along n-type phosphorus-doped mc-Si ingots.

ORAL

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6A.3

ORAL

Influence of Crucible Coating and Seeding Material on the Inclusions in Si Ingots from Directional Solidification

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Silicon (Si) wafers from directional solidification processes are actually the basis for around half of all sold solar cells. SiC or Si_3N_4 particles formed in the melt can be incorporated in the growing crystal and cause manifold problems such as mechanical stress in the crystal lattice, sawmarks on the wafer surfaces or electrical shunting in the final solar cell. Furthermore they can act as seeding points for new grains during growth[1].

Inclusions in Si bricks can be quantitatively measured by infrared transmission measurements. In our study of bricks from G1 and G2 sized Si ingots we analyzed the spatial distribution of inclusions down to 18 µm in size. A strong influence of the crucible coating on the amount of inclusions is observed. By using an adapted coating the amount of inclusions could be drastically reduced (see Figure 1).

Furthermore the seeding material for growth processes such as High Performance multicrystalline Silicon or Quasimono-Si has a strong influence on the inclusion distribution. An increase of inclusion concentration with seed size is observed (see Figure 2). As the initial melting process introduces Nitrogen from the coating into the melt a part of it recrystallizes in the spacings between the seed material leading to a reduction of inclusions in the grown bulk material.

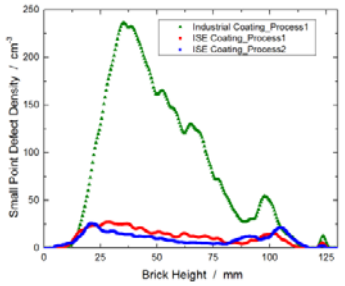

Figure 1: Small Point Inclusions over brick height for different crucible coatings

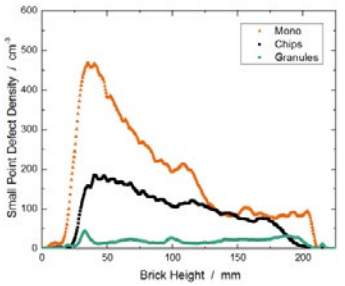

Figure 2: Small Point Inclusions over brick height for different Si seed materials

By identifying sources and validating their effect, we show possible ways of reducing harmful inclusion in Si from directional solidification such as an adapted crucible coating and an optimized seed configuration.

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7A.1

Simulation of Complex Rate Equations in Laser Crystals

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Multi-doped crystals as gain medium can improve the efficiency, output power and other physical properties of a laser. However, the design and test of a new laser system with a novel laser crystal is very time consuming. The simulation of a complete laser system can help to reduce this development time. Furthermore, simulations can avoid time consuming experiments. However, in order to obtain accurate simulation results, complex numerical simulations are needed. In particular, the excitation of electrons in solid-state gain media have to be calculated accurately. To this end, we present a new simulation technique, which extends the dynamic mode analysis (DMA) to a general system of rate equations. This technique allows a 3-dimensional simulation of excited states and an approximation of the laser beam by Gaussian modes. Furthermore, this novel simulation technique allows to model physical effects such as upconversion and cross relaxation. As an example we present simulation results for a laser system including a multi-doped Cr:Tm:Ho:YAG crystal, which exhibits strong interionic energy transfer processes.

INVITED

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7A.2

ORAL

Electronic aspects of adsorption at semiconductor surfaces: adsorption energy, equilibrium pressure and growth.

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Adsorption at semiconductor surfaces entail drastic change of their electronic properties: the number of surface states, their energy and occupation. During the process the electron may be transferred, decreasing or even increasing its energy which contributes to the overall energy balance, i.e. it affects the adsorption energy [1]. The electronic contribution may change the adsorption energy by several electronvolts [1]. The effect exists for the Fermi level pinned by surface states. For the Fermi level not pinned, for specific coverage, the sudden change of the adsorption energy occurs which is associated by the increase of the equilibrium pressure by several orders of magnitude. The typical growth proceeds in the latter situation i.e. Fermi level not pinned, being dependent on the Fermi level in the bulk, i.e. on the doping. The analyzed cases include hydrogen, ammonia and nitrogen at GaN(0001) and AlN(0001) surfaces [2,3]. The same results were obtained for hydrogen at nitrogen terminated GaN(000$\underline{1}$) surface [4]. Similar results are shown for Si adsorption at SiC(0001) Si-polar surface [5]. Thus the effect is universal, it occurs for majority of the growth processes, such as step motion, creation of multisteps during growth, etc. The electronic properties may be easily affected by presence of other species at the surface, affecting the electric charge balance. Thus the change of the surface electronic properties explains surfactant influence on the growth and doping of semiconductor crystals and layers.

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7A.3

A kinetic Monte Carlo model to compute homo-epitaxial growth of Ga_2O_3

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Ga_2O_3 has become a material of great interest for applications in high power devices [1]. Bulk crystals of β-Ga_2O_3 can be grown from the melt enabling wafer production for homoepitaxial layer deposition by MOVPE [2]. Depending on the miscut angle of the substrate, different surface structures have been observed during layer growth [3]. Our aim is to understand the growth kinetics in more detail and subsequently to be able to tune the process in the desired way. Such an understanding can be gained by a joined effort in characterization and numerical calculations.

Kinetic Monte Carlo (KMC) calculations provide the possibility to study the growth kinetics on atomic level. Here we present a KMC approach to cover the details of adsorption, diffusion, and desorption for the growth of β-Ga_2O_3 on the (100) plane of β-Ga_2O_3. The setup of KMC includes the crystallographic structure with the individual positions of Ga and O atoms. From TEM investigations it is known that the terraces are always a (100)-B plane [4] which is one of the cleavage planes. For miscut angles between 4° and 6°, depending on the growth conditions, step-flow growth was observed. For smaller miscut angles or a flat surfaces a growth of 2D islands, which are elongated towards the [010] direction, was observed. This has to be taken into account when defining the first set of rules for adsorption and diffusion. Preliminary results are presented and discussed.

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7A.4

ORAL

Lumped parameter model for silicon crystal growth from a granulate crucible

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At the Leibniz-Institut für Kristallzüchtung a process for silicon crystal growth from melt in granulate crucible (Si-GC) is developed. The crystal is pulled from an inductively heated melt pool in a solid silicon “self-crucible” that stabilizes in a bed of silicon granules.[1,2]

For the experimental development of a stable growth process and identification of suitable growth parameters, an analytical lumped parameter model was developed.[3] This model is also the first step towards a model-based control system for the process.

Unknown parameters of this model were numerically fitted to experimental data by optimization. The model was numerically solved for various growth conditions and the simulation results show a good agreement to the experimental data. An example of the simulated crystal radius for a crystal grown with steps in the crystal pulling rate, see Fig. 1, can be seen in Fig. 2.

Fig.1 Grown crystal from Si-GC experiment

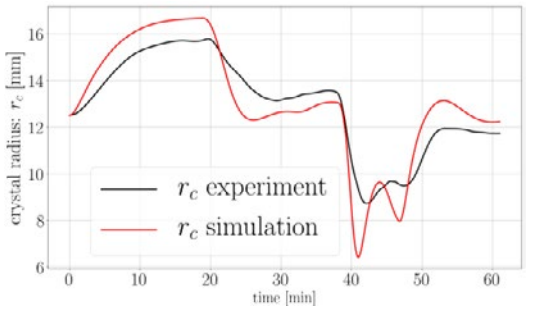

Fig. 2 Results of numerical simulation for the crystal radius in comparison to experimental data

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8.1

Challenges and Opportunities for Innovative Crystalline Materials in Europe – an IKZ perspective

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The mission of the "Leibniz-Institut für Kristallzüchtung (IKZ)" in Berlin is to be positioned among the internationally leading state-of-the-art competence center for science & technology as well as service & transfer for innovations *in* and *by* crystalline materials. To accomplish this mission, its R&D strategy ranges from basic (e.g. quantum materials, 3D crystals by 3D printing etc.) over applied research (e.g. rare-earth doped oxide and fluoride crystals for lasing, Aluminium nitride for UV technologies etc.) up to pre-industrial developments (e.g. Float zone 8" Si tool, Kristmag VGF GaAs etc.). Its research is in high demand, as crystalline materials are key enabling technology components to provide electronic and photonic solution for today´s challenges in society like artificial intelligence, energy, health as well as metrology.

Traditionally, IKZ is well established to provide innovations in crystalline materials by its combined in-house expertise on plant engineering, numerical simulations and crystal growth to achieve highest quality crystalline materials with tailored properties. Nanostructures, thin films and volume crystals are investigated with the latter being the unique selling point of the institute. A strong theoretical and experimental materials science (including modern synchrotron research) is a strong asset for IKZ´s R&D activities.

In future, together with partners (i.e. institutes & industries), the institute will dedicate more forces to drive innovations by crystalline materials, namely the reliable evaluation and benchmarking of innovative materials for disruptive technologies. Here, the future Horizon Europe Frame program as well as the German BMBF High Tech Strategy 2025 provide in future opportunities to establish academic – industrial platforms for common R & D.

Last but not least, education and teaching on all levels – from pupils over students up to senior scientist levels – is of central importance to set up and promote career pathways for common success in science & technology on crystalline materials.

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INVITED

8.2

INVITED

Organic molecules adsorption and ordering on the reconstructed crystalline surfaces

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Development of molecular electronics involves the use of monolayers and nanowires created by particles of organic compounds and/or individual molecules, as active components of nano-electronic devices. However, these molecular systems need some crystalline semiconducting or metallic substrates as a base to create functional electronic devices as switches, sensors, etc. Periodic structure of the substrate, surface reconstructions or defects may serve as pinning points for molecular systems and may strongly influence their electronic properties. Scanning tunneling microscopy (STM) is one of the most suitable technique to analyze the atomic or molecular structure of substrates and created nanostructures. Their electronic and chemical properties may be characterized by tunneling spectroscopy (STS) methods even with a subnanometer spatial resolution.

At first, results of STM/STS characterization of metallic and semiconducting substrates will be presented. The second part will be devoted to results on adsorption of individual organic molecules on metallic and semiconducting surfaces creating molecular switches and electron induced switching from trans to cis configuration will be reported[1,2]. Finally, the method of molecular chains growth in a controlled way on reconstructed metallic substrates will be shown[3].

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8.3

Floating zone growth and characterization of topological materials

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AWARD

With the direct access to a neutron source at PSI, the necessity for large bulk single crystals with a controlled stoichiometry is high. The travelling solvent floating zone technique is the best realization for this, as it is crucible free and enables in our setup with both argon and oxygen gas the appropriate atmosphere and pressures of 0-150 bar for the desired samples. In my talk, I will present two strikingly different systems of an oxide material, which is a spin dimer candidate as well as a magnetic Weyl semimetal. After a quick introduction to the method, the difficulties of the growth of (Ba,Sr)$CuSi_2O_6$ [1] will be discussed and I prove that due to the Sr introduction we now have the ideal quadratic dimer lattice to study the Bereshinski-Kosterlitz-Thouless phase by neutron diffraction and other techniques. The second system is CeAlGe, which with its variable stoichiometry gives a challenging candidate as even slight changes have a strong influence on its physics [2]. On stoichiometric crystals we find unlike promised by theory a complex incommensurately modulated magnetic groundstate below its antiferromagnetic like transition at 4.2 K.

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8.4

AWARD

Plasma-assisted MBE growth and properties of GaN nanowires

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It is well established that GaN nanowires (NWs) are promising building-blocks of new electronic and optoelectronic devices. Much effort is recently concentrated on controlled growth of GaN NWs on non-crystalline substrates, which offers a large degree of freedom in designing new devices. However, due to the lack of fundamental growth studies, the growth mechanisms underlying the formation of GaN NWs on non-crystalline substrates are still not well understood, hindering the control of the growth process.

Aim of this work is to show how the *in-situ* growth monitoring tools (RHEED, line-of-sight QMS) can be efficiently used to study nucleation and further axial growth of GaN NWs by plasma-assisted MBE (PAMBE). For that catalyst-free growth of GaN NWs is studied on Si with amorphous Al_xO_y (a-Al_xO_y) buffer deposited by atomic layer deposition [1]. Then, the results are compared with those obtained on standard nitridated Si substrates (Si_xN/Si) under the same growth conditions. This leads to a quantitative description of the nucleation and growth of GaN NWs by PAMBE on amorphous substrates [2, 3]. The growth process is analyzed *in-situ* from the incubation and nucleation stages till the formation of the final NW ensemble. We observe qualitatively the same temporal evolution of RHEED [2] and QMS signals [4, 5] for the two types of substrates. However, on a-Al_xO_y, the incubation time of 3D GaN islands is much shorter and the nucleation faster than on nitridated Si. Theoretical analysis of RHEED-measured incubation times indicates dominant heterogeneous nucleation mechanism on a-Al_xO_y while less efficient, homogeneous nucleation prevails on Si_xN [3]. Dedicated samples are used to analyze the evolution of surface morphology as the growth proceeds. In particular, a shape transformation from spherical cap-shaped GaN islands to the final NW-like geometry is found at the early stages of the growth on both substrates [4]. The effect explains why perfectly-aligned GaN NWs are formed perpendicularly to the substrate surface despite its amorphous nature. All the similarities found in this work for experiments on a-Al_xO_y and Si_xN/Si imply that this type of NW formation is not limited to a particular substrate but being general in nature is inherent to GaN growth by PAMBE, so should be transferable to numerous types of substrates.

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8.5

AWARD

Growth and optical properties of $SrTiO_3$

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Strontium titanate ($SrTiO_3$) is one of the most commonly used substrate materials for the epitaxy of perovskite thin films. However, advanced applications are limited by the high dislocation density (> $10^6/cm^2$) and by high mosaicity values (300 - 480″) of the currently commercially available Verneuil grown crystals. We have shown that high quality bulk crystals can be grown by top-seeded solution growth (TSSG) from self-flux[1]. Also, a defect minimized and virtually mosaicity-free $SrTiO_3$ single crystal was obtained through self-flux growth and will be presented here[2].

Self-flux grown $SrTiO_3$ crystals and single crystal fibers produced by the laser heated pedestal growth (LHPG) method show colorations that depend on the oxygen concentration in the growth atmosphere[1,3]. Low oxygen concentrations cause many oxygen vacancies in the crystals, which lead to a high free electron concentration and a blue/black color. Oxygen rich atmospheres cause a brown crystal coloration, which was investigated in stoichiometric TSSG crystals. A combination of TEM and Mie-scattering calculations showed that nanometer sized voids cause the brown color[5]. It is proposed that these nano-voids form through vacancy clustering. Far fewer nano-voids are formed in oxygen poor atmospheres because the cation vacancy formation energy is much higher under these growth conditions[6].

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9A.1

INVITED

Investigations on the growth of $CuAlO_2$ substrate crystals

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The interest in transparent, conductive and semi-conductive materials for oxide electronics (so called Transparent Conductive Oxides-TCOs) strongly raised up. $CuAlO_2$ –Delafossite (ABO_2) shows p-type behaviour and a relatively high mobility of holes due to hybridization of oxygen p-orbital with Cu^{1+} d-orbital [1]. The structure consists of two alternating layers, a planar layer of Cu-cations in a triangular pattern, responsible for p-type, formed with the above and underlying oxygen O-Cu-O dumbbells and a layer of edge-shared AlO_6 octahedra which are flattened with respect to the c-axis [2]. It has an indirect bandgap of 2.22 eV and a direct one of 3.4 eV [3].

So far $CuAlO_2$ has only been grown as polycrystalline thin layers or small crystallites. However, active devices based on single crystalline layers of high structural quality require single crystalline native substrates. For their applications in transparent p-type semiconductors, the single crystals should have high transparency and conductivity.

For the top-seed solution growth (TSSG) technique with a Czochralski-setup, a suitable crucible material has to be found that does not react with either copper or its oxides in the melt. As alternative, for a crucible-free growth, the optical floating zone (OFZ) technique is preferred.

For an understanding of the crystallization of $CuAlO_2$, the existing Cu_2O-Al_2O_3 phase diagram proved to be insufficient. Firstly, under sufficiently oxidizing atmospheres, most melting and crystallization processes are accompanied by chemical redox reactions between Cu_2O and CuO. On the other hand, if the oxygen activity in the growth atmosphere is too low, metallic copper can be formed in platinum crucibles, due to the entropic stabilization of Cu by alloying with Pt [4].

DTA/TG measurements have been conducted to explore the phase diagram and solution growth conditions of $CuAlO_2$. The investigations revealed that solution growth should be carried out with a solvent composition of 1-2 mol% Al_2O_3 in Cu_2O. These results enabled the growth of $CuAlO_2$ crystals in Cu_2O rich melt solutions [4].

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9A.2

ORAL

A new class of amino acids salts

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Amino acid salts are a source of promising nonlinear optical, pyroelectric, ferroelectric materials, and they are also used in medicine[1]. Salts with the same amino acid of various types (A^+X^-, $(A^+)_2Y^{2-}$, $(A^+\cdots A)X^-$, $A^+(A^+\cdots A)Y^{2-}$, $(A^+\cdots A)_2Y^{2-}$, where A^+ and A are amino acids in singly charged and zwitterionic state, respectively, X^- and Y^{2-} singly and doubly charged anions) as well as mixed salts with different anions of various types are known[1]. Recently we have discovered a whole new class of salts containing different amino acids. To date, more than 40 similar salts of various types are obtained. Some types have analogs among salts with the same amino acid. *E.g.*, the salts (β-AlaH)(BetH)$SiF_6{\cdot}H_2O$ and (β-AlaH)(*L*-ProH)SiF_6 of the type $A(1)^+A(2)^+Y^{2-}$ are analogs of the $(A^+)_2Y^{2-}$ type. The salts (β-AlaH)(*L*-ProH···*L*-Pro)SiF_6 and (BetH)(BetH···Sar)$SiF_6.H_2O$ ($A(1)^+[A(2)^+\cdots A(2)]Y^{2-}$, $A(1)^+[A(1)^+\cdots A(2)]Y^{2-}$ types) are analogs of the $A^+(A^+\cdots A)Y^{2-}$ type. The majority of obtained salts belongs to the $[A(1)^+\cdots A(2)]X^-$ and $[A(1)^+\cdots A(2)]_2Y^{2-}$ types. Salts with dimeric cations (GlyH···Sar), (GlyH···DMG), (DMGH···Sar), (BetH···Sar), (BetH···DMG), (β-AlaH···Sar), (β-AlaH···DMG), (β-AlaH···Bet), (β-AlaH···*L*-Pro), (*L*-ProH···Sar) and various anions were also obtained. Some other salts (*L*-ArgH)(Sar)Cl, (*L*-ArgH)(Sar)Br, (*L*-ArgH)(Sar)I, (*L*-ArgH)(DMG)I, (*L*-ArgH)(Bet)Cl, (*L*-$ArgH_2$)$(DMG)_2I_2$, (*L*-$ArgH)_2$(GlyH···Gly)$I_3.H_2O$, (*L*-$ArgH)_2$(β-AlaH···β-Ala)I_3 can be considered as combinations of different types. More than half of the salts obtained are non-centrosymmetric, 16 of which have polar symmetry. Thus, these salts are potential nonlinear optical and pyroelectric crystals. Additionally, the iodides can be used as regulators of thyroxine synthesis in the body[2].

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9A.3

ORAL

Flux crystal growth of ErV_2Al_{20} and related $CeCr_2Al_{20}$-type aluminides

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We have grown single crystals of ErV_2Al_{20} intermetallic cage compound and some related $CeCr_2Al_{20}$ aluminides by using an Al self-flux method employing a setup with two crucibles and alumina frit-disc for flux separation.

Magnetization of ErV_2Al_{20} phase shows Curie-Weiss paramagnetic behavior down to 1.9 K. Heat capacity and resistivity measurements have revealed the influence of a large-amplitude, low-frequency anharmonic “rattling” mode which can be ascribed to Er atoms positioned within supersized Al cages [1].

Flux growth technique used for ErV_2Al_{20} was also successfully applied to LaV_2Al_{20} superconductor. It was found that in case of late lanthanides, such as Tm or Lu a hexagonal $Ho_6Mo_4Al_{43}$-type phase is obtained instead of the targeted cubic $CeCr_2Al_{20}$.

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10A.1

Single crystal growth and electronic transport properties of topological semimetals

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INVITED

Initiated by the discovery of topological insulators, topologically non-trivial matter, especially topological semimetals (TSM), has emerged as a new frontier in the field of quantum materials. Among various TSMs, one recognizes Dirac semimetals (DSM) that exhibit accidental band touching between conduction and valence bands, protected by time-reversal and inversion symmetry, as well as Weyl semimetals (WSM), in which one of those symmetries is broken, and single-degenerated band crossings with linear dispersion possess non-zero chiral charge. Another subgroup of TSMs consists of nodal-line semimetals (NLSM) with Dirac band crossings forming a closed trajectory in momentum space, protected by mirror, glide, or screw symmetries. The presence of nearly massless quasiparticles near chemical potential gives rise to unique transport properties of TSMs, like ultra-high charge carrier mobility, non-trivial Berry phases, huge non-saturating magnetoresistance (MR), weak antilocalization effect, chiral magnetic anomaly (negative longitudinal MR, planar Hall effect, angular narrowing of longitudinal MR), etc. The most intriguing physical phenomena observed in TSMs not only provide excellent tests for fundamental theories, but also promise a wide range of possible applications in low-power spintronics, optoelectronics, quantum computing and green energy harvesting.

Here, we briefly review on our recent accomplishments in the blooming field of TSMs. We present our results in growing high-quality single crystals by means of suitable preparation techniques (Czochralski, Bridgeman, metallic fluxes, chemical vapor transport), our experimental observations of Dirac/Weyl states via angle-resolved photoemission spectroscopy (ARPES), and concisely account for our comprehensive characterization of the anomalous low-temperature electronic transport behaviors in several different DSM, WSM and NLSM materials.

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10A.2

ORAL

Characterization of doped and undoped single crystalline films of perovskites using Raman spectroscopy

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The perovskite-based materials are extensively studied for a few decades due to their great technological relevance, where their use as a host materials for rear earth elements gives widespread areas of applications. New perspective for perovskite materials appeared along with crystallization of single crystalline films (SCFs) by liquid phase epitaxy method (LPE). The use of different fluxes during the growth process allows to obtain layers of better luminescent properties. One of the most important and promising field of applications of SCFs of perovskites is scintillation technology[1-3].

Ce^{3+} doped single crystals (SC) of perovskites e.g. $YAlO_3$ (YAP) and $LuAlO_3$ (LuAP) have become prevalent for use as scintillators since the 1980's. During the last decade single crystalline films (SCFs) of Ce^{3+} doped $YAlO_3$ [2] and $LuAlO_3$ [3], as well as the Ce^{3+} and Mn^{2+} doped $TbAlO_3$ (TbAP) perovskites [4] were synthesized using liquid-phase epitaxy method onto YAP substrates.

In this paper we present Raman spectroscopy investigation of undoped YAP, Mn^{2+} doped YAP and TbAP and Ce^{3+} doped YAP and LuAP single crystalline films (SCFs) grown onto YAP substrates. The Raman spectra and Raman maps recorded for YAP:Mn@YAP, TbAP:Mn@YAP, YAP:Ce@YAP and LuAP:Ce@YAP epitaxial structures allow a distinction between SCF and the substrate and furthermore allow identification of a transition layer between SCF and the YAP substrate.

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Optical properties of $Cd_{1-x}Zn_xTe$ bulk crystals

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$Cd_{1-x}Zn_xTe$ [where 0<x<1] mixed crystals were grown from the melt by the modified high-pressure Bridgman–Stockbarger method. The elemental composition of samples was established by Quantax 200 X-ray spectrometer and EDX XFlash 4010 detector[1]. The Real (ε_1) and imaginary (ε_2) parts of dielectric constant were obtained using V-VASE ellipsometer (J.A.Woollam Co., Inc.) in energy range from 1.24 to 4.13 eV.

Luminescence spectra (Fig. 1.) were recorded in temperature range from 10K to 300K. These spectra showed temperature-dependent qualitative and quantitative changes in light emission. It was found that they depend on crystal composition.

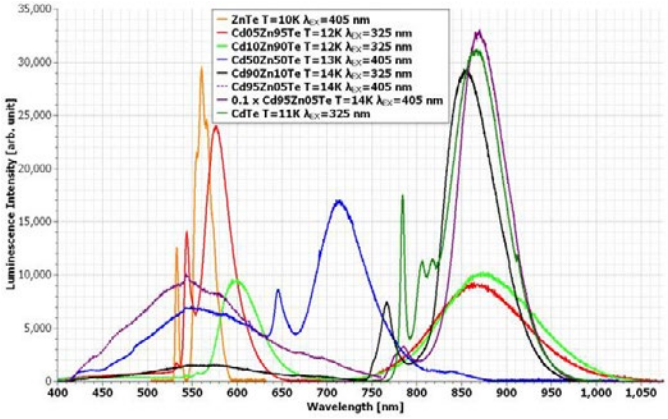

Fig. 1. Luminescence spectra of $Cd_{1-x}Zn_xTe$ crystals excited with λ=325nm (P=25 mW) or λ=405nm (P=50 mW) at temperature range (T=10-14K)

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ORAL

1B.1

Growth of large single crystals of a new quantum spin liquid compound $Ca_{10}Cr_7O_{28}$

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INVITED

Quantum spin liquid is a highly sought after exotic state of material in which static magnetism is absent due to coherent motion of spins down to the lowest temperature. $Ca_{10}Cr_7O_{28}$ in one of a very few material where experimental realizations of a spin liquid was discovered [1]. However growth of single crystal of $Ca_{10}Cr_7O_{28}$ is quite challenging due to several reasons, such as, mixed valence states of the Chromium ions, incongruent melting of the compound, instability of the desired phase below a certain high temperature, etc. In this talk, I will present how we overcame these challenges to grow high quality single crystal by the Traveling-solvent-floating-zone technique in an optical floating zone machine.

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Absolute Up-conversion Quantum Yield of SrF_2:Yb^{3+}, Er^{3+} Single Crystals

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Most luminescent materials exhibit a Stokes-shifted or downshifted emission with the emitted photons having lower energy than the absorbed ones. Few materials possess the ability to generate anti-Stokes shifted photoluminescence or upconversion (UC). In the last case, the emitted photons have higher energy than those used for excitation. The phenomenon of UC is particularly interesting from point of view strong application potential for different fields of science and technology including 3D displays, thermometry, solid state lighting, lasers, solar energy harvesting, sensors, bio-imaging, drug delivery, as well as plastics recycling and anti-counterfeiting [1].To better determine the UC properties of phosphors and to lay the foundation for their application, the single crystal of these phosphors is a very attractive material. Perhaps, the UC single crystals are the simplest starting point system in a long avenue of UC research with several structural parameters, which can be well controlled (phonon energy, symmetry of crystal structure, doping concentration).

In our work we investigated UC luminescence of SrF_2:Yb^{3+}, Er^{3+} materials in order to answer the question: can alkaline earth fluoride hosts doped with Ln^{3+} ions compete with the most efficient UC materials? In order to answer the question, we grew single crystals based on SrF_2 host doped with different concentrations of Yb^{3+}/Er^{3+} pairs of ions, investigated their UC spectral UC properties and determined their UC quantum yield (UCQY). The highest UCQY of 6.5 % was measured for the crystal co-doped with 2 mol. % Er^{3+} and 3 mol. % of Yb^{3+}. The obtained results were compared with properties of sub-micron powders of SrF_2:Yb^{3+}, Er^{3+} with the same chemical composition [2].

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1B.3

ORAL

QD-based luminescent nanocomposite glass produced using the NPDD method

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NanoParticle Direct Doping (NPDD)[1] is a method developed in the Institute of Electronic Materials Technology in Warsaw that allows fabrication of volumetric composites based on glass matrices doped with various kinds of nanoparticles, including Quantum Dots (QDs).

In this work we attempted to obtain photoluminescent (PL) materials doped with one or two different types of QDs, varying in size and composition. As the matrix we have chosen the sodium borophosphate dielectric glass (NBP), which is transparent over wide range of wavelengths, and exhibits melting temperature of ca. 750 °C.[2] As optically active elements we have used variety of QDs: CdSe/ZnS core-shell QDs, CdTe QDs, ZnCdSeS QDs, as well as cadmium free Zn-Cu-In-S/ZnS core-shell QDs; all of them showing luminescence at visible light.

We have prepared series of samples. Absorbance and PL spectra were measured to characterize the materials, as well as time-resolved fluorescence lifetime measurements to study the process of energy transfer between different kinds of quantum dots.[3]

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1B.4

Enhancing luminescent properties of doped glasses with plasmonic nanoparticles

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Combining quantum dots with plasmonics results in strong exciton-plasmon interactions leading to a fast and intense luminescence that can be applied in novel optical devices. However, demonstration of such coupling in a volumetric material poses a challenge and has been mostly investigated at the nano- or microscale systems[1,2]. Here we present a volumetric nanocomposite made of glass simultaneously doped with both CdTe quantum dots (CdTe QDs) and silver nanoparticles (Ag NPs) fabricated by NanoParticle Direct Doping method (NPDD)[3]. The material exhibits ultra-fast (90 ps), narrow (13 nm) and amplified photoluminescence at 503 nm. Properties of the emission, i.e. decrease of PL lifetime by two orders of magnitude compared to the glass material only with quantum dots, confirm that emitting QDs are coupled to nanocavities between two plasmonic nanoantennas. Easy fabrication of bulk plasmonic-excitonic composites builds a bridge between laboratory research and real-life applications, such as high-performance lasers or new types of biosensors.

ORAL

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2B.1

INVITED

Germanium doping of GaN in MOVPE

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GaN is the commercially second important semiconductor after Si enabling LEDs and blue lasers but also transistors for power and microwave devices. For most optoelectronic devices doping of GaN is essential to enable current flow. While Si has long been the only established option as an electron donor in GaN, germanium nowadays turns into the focus because of higher doping levels that can be achieved in MOVPE. I will give an overview on the differences of Ge and Si doping of GaN and point out the benefit for device structures as well as challenges in MOVPE growth.
In particular Ge-doping eases the realization of tunneling contacts and can act as transparent conductive nitride, e. g., replacing ITO contacting and current spreading layers in LEDs. But also GaN based devices as vertical cavity surface emitting lasers (VCSEL) can benefit from highly conductive and transparent GaN:Ge layers.

Due to the high electron concentration that can be achieved and the Burstein-Moss-shift that is induced by it GaN:Ge has a relatively large change in refractive index in contrast to low n-doped GaN enabling a homoepitaxial distributed Bragg mirror which is useful for narrowband wavelength stabilized LEDs.

Lastly even a new physical quasiparticle the collexon, leading to an exciton like emission at doping levels in the mid to upper 10^{19} cm^{-3} range, was observed. The formation of this quasiparticle is believed to be due to the near to ideal behavior of Ge as dopand when replacing Ga which has a nearly identical ionic radius and binding energy.

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2B.2

Influence of hydrogen on GaInN growth in Metalorganic Chemical Vapour Phase Epitaxy

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Nitride semiconductors (AlGaInN) have created multibillion markets of white LEDs and of blue/green laser diodes for Bluray, data-transfer-rate in the LiFi communication, RGB television with a superb colour resolution and 3-dimensional projection without goggles. Nitride LDs will be also the base for creating many niche markets like quantum technologies (atomic clocks, mass sensors, cryptography, etc), welding of copper and gold, in medicine, military applications, and many others.

However, despite the commercial successes (present and future) many properties of nitride semiconductors are not know and understood. One of the most important and still controversial issues in nitride epitaxy is an application of hydrogen as a carrier gas in the MOVPE technology. For other III-V semiconductors, hydrogen is commonly used as a carrier gas because it removes oxygen and makes the surface and interfaces smoother. However, it cannot be used for growing InGaN quantum wells (the active part of LEDs and LDs) because it prohibits indium incorporation.

In the first part of our presentation, we will show that hydrogen not only removes indium from the InGaN layer, but also passivates the surface and indium cannot be incorporated.

In the second part, we will present the optimization steps to achieve the best InGaN QWs properties for LD manufacturing. These steps include the optimization of hydrogen used in the carrier gas and the time of growth-breaks between the QWs and QBs.

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ORAL

2B.3

ORAL

LPE method as a useful tool for development of the composite luminescent materials based on the mixed garnet compounds

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The report presents the last achievements of our group at IF UKW in the development of the luminescent materials based on the single crystalline films (SCFs) and single crystals (SCs) of mixed garnets using the liquid-phase epitaxy (LPE) method for application as scintillating materials for radiation monitoring and microimaging as well as the phosphors for photo-conversion (pc) in white LEDs.

The two mainstreams of the research are considered:

1. Development of multilayer composite scintillators (CSs) based on SCFs and SCs of mixed garnets for simultaneous registration of different types of ionizing radiation (α-, β-particles and γ-quanta) and microimaging with spatial resolution in the submicron range. Such CSs have been made in the form of multilayer epitaxial structures containing one or two scintillators in the form of SCFs grown the LPE onto substrates from SC scintillators. The SCs and SCFs parts of CSs have been fabricated from the effective scintillation materials based on the Ce^{3+} doped simple and mixed $A_3B_5O_{12}$ garnets, where A= Lu, Gd, Tb; B= Al, Ga with signficantly different luminescent spectra or scintillation decay kinetics.

The report presents the results on fabrication of scintillators based on the mentioned garnets in the form of SCs, SCFs and CSs and the investigation of their scintillation properties. The results of testing of the developed CSs in the detectors for radiation monitoring of mixed ionizing fluxes and the scintillating screens in microimaging technique will be presented as well.

2. Development of pc-converters based on SCFs and SCs of mixed garnet compounds for high-power $WLED_S$. All solid state pc-converters can be produced in the form of multilayer epitaxial structures containing film converters grown by the LPE method onto substrates from SC converters with different excitation and luminescence spectra. Such types of hybrid pc-converters have been produced from novel and effective luminescent materials on the basis of Ce^{3+}, Eu^{3+} and Mn^{2+} doped mixed garnets with general formula $\{A^{I}_{3-x}A^{II}_{x}\}B^{I}_{2-y}B^{II}_{y}](C^{I}_{z}C^{II}_{3-z})O_{12}$, where A= Y, Lu, Gd, Tb, Ca; B=Al, Sc, Ga, Mg; C=Al, Ga, Si, grown by the LPE method in SCF form onto undoped and Ce^{3+} doped of YAG and other garnet substrates.

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3B.1

Growth, structural peculiarities and optical properties of rare earth-doped gadolinium gallium aluminum garnet crystals

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INVITED

Solid solution $Gd_3(Al,Ga)_5O_{12}$ (GAGG) crystals with nominal stoichiometry $Gd_3Al_{2.5}Ga_{2.5}O_{12}$ or $Gd_3Al_2Ga_3O_{12}$ undoped, single-doped with trivalent rare earth ions or containing Yb^{3+}+ RE^{3+} pairs (RE =Pr, Ho, Er, Tm) were fabricated by the Czochralski method. Starting oxides were first dried at 1000 ^{0}C for 4 hours before weighting. Next, powders in appropriate molar ratios were mixed together and then pressed into cylindrical pellets under pressure of 200 kPa and calcinated at 1350 ^{0}C. Single crystals were grown under nitrogen atmosphere on <111> oriented seeds with a pulling rate of 2.5 mm/h and a speed of rotation 20 rpm. Transparent crystals 20 mm in diameter and 60 mm in length were grown with a convex crystal-melt interface from an inductively heated iridium crucible 40 mm in diameter. Low temperature spectroscopic study of obtained crystal samples provided a rich set of experimental data. They made it possible to propose some useful generalizations regarding structural disorder induced by a partial substitution of aluminum by gallium ions and location of incorporated luminescent rare earth ions. In particular, it was ascertained that resulting differences in crystal field give rise to marked inhomogeneous broadening of spectral lines and create non-equivalent sites. Derived energies and crystal field splitting of multiplets combined with excited state relaxation dynamics made it possible to determine energy gap law that governs rates of multiphonon relaxation of rare earth ions in GAGG host. Mechanisms involved in observed down- and up-conversion phenomena were proposed based upon these data. Ability of Ho^{3+}, Er^{3+}, Tm^{3+} and Yb^{3+} ions to show infrared laser operation in GAGG matrix was thoroughly examined and spectroscopic parameters relevant to this purpose were determined.

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3B.2

ORAL

Ternary phase diagram studies and single crystal growth of solid solutions in the Ga-Pd-Sn system for basic research in heterogeneous catalysis

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Recent studies have shown, that intermetallic compounds may have better catalytic properties than presently used industrial catalysts that are based on elements or substitutional alloys[1]. Those allow diffusion and segregation under operation conditions, while intermetallics have structures with well-defined atomic sites. Therefore, they may have better long-term stability and considerably higher selectivity, according to the *active site isolation concept*[2].

The intermetallic compounds GaPd and $GaPd_2$ have been found to be promising candidates for heterogeneous catalysis in hydrogenation processes with Pd being the active element. Both compounds have already been grown with the Czochralski technique from Ga-rich solution in cm^3-sized single crystals[3], which are required for fundamental studies in surface physics and crystallography.

Both, $GaPd_2$ and $SnPd_2$ crystallize in the Co_2Si structure type (*Pnma*) and form a complete solid solution series. Therefore, Ga substitution by Sn in $GaPd_2$ is a powerful approach to discriminate and understand catalytic properties arising from either structural or electronic influences. Pure $SnPd_2$ cannot be grown from a liquid phase, but decomposes in a peritectoid reaction at 820°C[4].

Due to first successful experiments on $Ga_{1-x}Sn_xPd_2$ the substitution of Ga by Sn in GaPd also came into focus. The end members GaPd ($P2_13$) and SnPd (*Pnma*) can be grown from solution, but no complete miscibility is present.

This study presents first results of single crystals of $Ga_{1-x}Sn_xPd_2$ and $Ga_{1-x}Sn_xPd$ grown by the Czochralski technique from (Ga,Sn)-rich solution based on a high number of simple cooling pre-experiments in order to investigate the relevant tie-lines the ternary Ga-Pd-Sn system.

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3B.3

Modification of micro-pulling-down apparatus for crystal growth of cesium hafnium chloride by Bridgman method

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ORAL

Newly developed micro-pulling-down (μPD) method is an excellent method for so called materials' screening as an important tool in the optimization of material properties. Its main advantages consist in the fast crystal growth and low material consumption making it highly cost effective, when compared to traditional growth techniques such as Czochralski and Bridgman methods. Regardless of those advantages some materials are difficult to grow by the standard μPD setup.

Recently discovered cesium hafnium chloride Cs_2HfCl_6 (CHC), identified by Burger et al.[1] as a promising scintillator for gamma spectroscopy, belongs into group of materials uneasy to growth using standard μPD. CHC is formed by reaction of CsCl and $HfCl_4$ in stoichiometric ratio (2:1) heated to molten state. It crystalizes in cubic structure with lattice parameter of $a = 10.42 \pm 0.01$ Å.[2] However, the preparation of CHC and its subsequent crystal growth is complicated due to low sublimation temperature (at ca. 320 °C) and high hygroscopicity of $HfCl_4$. This problem can be solved by growing the crystal in an enclosed system.

In this contribution we present a newly developed technique for crystal growth of CHC using the μPD apparatus modified for the vertical Bridgman growth in sealed quartz ampules. This setup allowed the growth of undoped CHC crystals with ca. 7 mm in diameter and 40 mm in length with sufficient crystal and optical quality for further evaluation of scintillation performance. Basic material properties such as phase purity, crystal quality, and luminescence performance will be presented.

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3B.4

ORAL

Crystal growth of cesium hafnium chloride by Bridgman method, its stability and luminescence and scintillation properties

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Newly discovered cesium hafnium chloride Cs_2HfCl_6 (CHC) is considered (due to its excellent scintillation properties) as a promising candidate for cost effective radiation detectors.[1] The CHC is formed by CsCl and $HfCl_4$ mixed together in stoichiometric ratio 2:1. It melts congruently at 826 °C and crystalizes in cubic crystallographic structure.[2] The CHC is considered as relatively stable with low hygroscopicity, but still reaction with moisture when exposed to air atmosphere was reported.[3] In this contribution we were focused on the preparation and crystal growth of CHC by vertical Bridgman method. Samples of various dimensions were cut and polished for characterization of optical, luminescence, and scintillation properties, and their decay kinetics. Experiments of the CHC stability when exposed to ambient atmosphere were performed. Optical microscopy allowed examination of the optical quality of grown crystals. The influence of the crystal quality and sample size on the scintillation performance was evaluated as well.

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4B.1

Influence of grain size on optical properties of RE-doped nanocrystals

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INVITED

The blue emitting phosphor Sr_2CeO_4, prepared by combinatorial route in 1998, attracted much attention due to its exceptional optical properties such as intense broad absorption as well as emission bands in visible range under UV excitation resulting from charge transfer transition between metal and ligand or relatively long decay times (~50μs)[1]. These features combined with good chemical stability and high luminescent efficiency gives many possibilities for industrial applications.

Here we present synthesis method and spectroscopic characterization of the pure Sr_2CeO_4 and RE^{3+} doped nanocrystals in terms of size effect. The fine powders were prepared using modified sol-gel method and annealed in temperature range of 750-1050°C.

X-ray diffraction patterns confirmed phase purity of the samples calcined above 800°C. The impact of size effect on luminescent properties was investigated. In particular the absorption, excitation and emission spectra as well as the emission kinetics of the pure Sr_2CeO_4 and RE^{3+} doped nanocrystals were studied.

Moreover, the anti-Stokes emission spectra of pure Sr_2CeO_4 and RE^{3+} doped nanocrystals were investigated under irradiation with focused beam of CW laser diodes in vacuum atmosphere [2,3]. It was found that under exceeding excitation threshold the intense broad band white emission was observed. The mechanism of white light emission will be discussed in terms of intervalence charge transfer within the (RE^{2+},RE^{3+}) ion pair.

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4B.2

ORAL

Metallic nanocrystals via an aerosol route

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Mass production of nanocrystals with accurate size control is a central problem in nanotechnology. Aerosol technology offers the possibility to produce nanocrystals with well-controlled composition and size distribution.

An aerosol is defined as solid or liquid particles suspended in a gas, and aerosol science and technology has been used since over 60 years, primarily to study the size, shape and composition of airborne particles [1]. For this purpose, tools have been developed, which allow scientists to fabricate and precisely classify particles in the micro- and nanometer size ranges according to size, and to measure their concentration in the carrier gas [2]. The aerodynamic properties of nanoparticles depend almost exclusively on their size and shape and only to a small extent on their mass and composition, which means that the same generic characterization and production tools can be applied to a variety of different materials [3].

Here, I will present a setup of aerosol tools and methods that can be used to generate well-defined metallic nanocrystals.

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Instruments for crystal growth and crystal characterization

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This presentation will provide an overview of three instruments suitable for crystal synthesis and physical characterization. Two furnaces and the Physical Properties Measurement Systems (PPMS) will be discussed.

Besides a well-known halogen-lamp based floating zone furnace for crystal growth a new, unique furnace equipped with lasers is presented.

The PPMS is a low temperature, high magnetic field platform for a wide range of physical measurements and can be used for example for electric, thermal and magnetic characterization.

5B.1

INVITED

Crystallographic and dielectric characteristics of $PbB'_{1-x}B''_xO_3$ single crystals grown by the flux method

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Lead based complex perovskites $PbB''_{1-x}B''_xO_3$ exhibit a great variety of physical behavior and, due to their excellent properties, are widely used as sensors, transducers and actuators. The variety of observed physical properties results from different states of the B-cation chemical order.[1,2] The driving forces and consequently the degree of order depend on differences between the ionic charges and radiuses of B-cations.[1]

Good quality single crystals of $PbIn_{1/2}Nb_{1/2}O_3$ (PIN), $PbIn_{1/2}Ta_{1/2}O_3$ (PIT), $PbYb_{1/2}Nb_{1/2}O_3$ (PYN), $PbYb_{1/2}Ta_{1/2}O_3$ (PYT), $PbMg_{1/2}W_{1/2}O_3$ (PMW), $PbFe_{1/2}Nb_{1/2}O_3$ (PFN), $PbFe_{1/2}Ta_{1/2}O_3$ (PFT) and $(1-x)PbIn_{1/2}Nb_{1/2}O_3$ –$xPbTiO_3$ (PINT) were grown by the flux method. The PbO-PbF_2-B_2O_3 system was used as a solvent.

Room temperature X-ray tests and broad temperature range dielectric and differential scanning calorimetry (DSC) studies were performed. For studied single crystals both normal sharp or diffused ferroelectric or antiferroelectric phase transitions were detected. The results of these investigations allowed us to connect different mature of phase transitions with the degree of the 1:1 chemical order of the B-ion sublattice.

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5B.2

Growth and characterisation of radiation sensors based on metal-organic lead bromide perovskite crystals

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Competitive nature of the hybrid methylammonium lead bromide perovskite $MAPbBr_3$ as an X-ray radiation sensor stems from its low-cost and low-temperature solution synthesis. Although a relatively fast and in principle simple synthesis, it yields single crystals with a performance comparable to those of the Cd(Zn)Te.[1] A systematic approach to crystal growth and characterisation has been used to address a significant lack of information on the crystal growth features and XRD crystal structure including the sparse data on the key electrical performance parameters in literature. The orange, semitransparent $MAPbBr_3$ crystals are up to 7 mm x 7mm x 2 mm and have even surfaces and a primitive cubic lattice. The samples grown in templates using the temperature-dependent growth methods[2,3] are monocrystalline, but have strained lattices. In contrast, the sample grown by the isothermal antisolvent vapour crystallisation (AVC) method[1] displays the most uniform growth features, has the lowest density of defects and consequently the most pristine crystal structure. All have the dominant lattice plane (200) roughly parallel to the sample surface. The graphite-$MAPbBr_3$ contacts exhibit the linear Ohmic behaviour. The electrical performance parameters determined from the Hall effect measurements in van der Pauw geometry agree with the published values: the change carrier concentration of 10^9-10^{10} cm^{-3}, the density of trap states of 10^9-10^{10} cm^{-3}, the resistivity of 10^7-10^8 Ωcm and the carrier mobility of 7-289 $cm^2V^{-1}s^{-1}$. All except the AVC grown crystal are the p-type semiconductors. The proposed optimal crystal growth involves the growth of a seed crystal on an O_2-plasma treated oriented substrate by the AVC method followed by the further growth of this crystal by the low-temperature gradient control method[3]. This systematic approach to the crystal growth can potentially yield a high performing $MAPbBr_3$ semiconductor material that satisfies the requirements for direct X-ray conversion radiation sensors.

ORAL

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5B.3

ORAL

Growth of H-Nb_2O_5 bulk crystals

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The oxides of niobium form a complex system consisting of numerous phases of different polymorphic forms. Being able to adopt different structures, niobium oxides possess interesting properties in science and technology. Beside the usage as solid electrolyte capacitor or complementary metal oxide semiconductor[1] current research studies are focused on the development of resistive switching devices for non-volatile memories and memristors [2], which in part rely on the availability of H-Nb_2O_5 single crystals. This contribution will give first insights in our growth activities of this high temperature phase using the optical floating zone technique. Due to the low symmetry (monoclinic) and metastability, a complex growth process optimization is required to fully eliminate the formation of cracks and twins, which are by far the most critical remaining issues. To improve the structural quality of the crystals the following growth parameters were varied so far: growth-rate, oxygen partial pressure, feed and seed rod preparation procedure, heating and cooling rates and the filament height of the lamps. Currently, our activities are focused on the preparation of high quality ⟨001⟩ seeds, which are after Shindo and Komatsu [3] most suited to obtain twin-free crystals. The potential in using alternative growth techniques will be also discussed, since single crystals of niobium(V)-oxide are not commercially available.

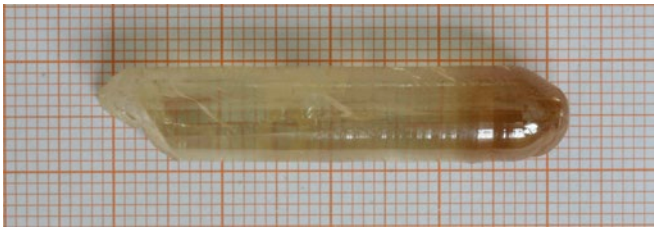

Fig. 1 (a) H-Nb_2O_5 crystal grown by using the optical floating zone technique.

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6B.1

Fluoride single crystals for solid-state laser applications

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INVITED

Solid-state lasers based on rare-earth-doped fluorides single crystals have been proven to be useful and reliable light sources. These materials gave access to laser emission in previously unexplored spectral region and are still competitive in the already covered regions thanks to their better output mode quality, enhanced spectral lifetimes, narrower emission linewidths, reduced sensitivity to electrical noise. Solid-state visible lasers can be employed in a wide range of applications in research, photonics, communication, remote sensing, and medicine [1]. Eye-safe sources around 2 μm, on the other hand, can be used in the realization of long-range LIDARs, laser scalpels, and gas detection in the atmosphere [2].

We present a review on our recent work on solid-state sources based on these materials, in the visible and near infrared regions. In particular, we focused on visible laser generation using Pr-doped materials and NIR laser generation, around 2 microns, using Tm or Tm-Ho co-doping.

We report on visible laser operation in Pr:$LiLuF_4$ (LLF), Pr:KY_3F_{10} (KYF), and Pr:$LiYF_4$ (YLF) [3], in the red, orange and deep red regions, comparing the well-known Czochralski growth technique with the more recent micro-pulling-down method, that allows to grow long cylindrical samples, with enhanced thermal dissipation, called single-crystal fibers (SCFs). We also present results on laser emission, power scaling, and tunability on other appealing fluoride such as Pr:BaY_2F_8 (BYF) and its isomorph Pr:$Ba(Y_{0.8}Lu_{0.2})_2F_8$ (BYLF) [4]. Among 2-micron laser materials, we present recent results on laser operation and tunability in Tm:SrF_2 [5], Tm:KYF, Tm-Ho:KYF, and other appealing materials, which showed remarkable performances in terms of slope efficiency and tuning range.

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6B.2

ORAL

Prospective active media for UV, Visual and IR spectral ranges on the basis of scheelite-type and colquiriite-type fluoride mixed crystals

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Laboratory of crystal growth in Kazan Federal University historically has great experience of high temperature methods of crystallization of doped fluoride crystals and transfer the technologies of their production. Our background numbers various types of compounds among which $LiYF_4$ and its homologous crystals, $LiCaAlF_6$, $LiSrAlF_6$, BaY_2F_6, LaF_3, CeF_3, KY_3F_{10} and many others.

Here we present and discuss our recent achievements in approach for improvement optical quality and segregation coefficient for rare-earth activators in compounds $LiY_{1-x}Lu_xF_4$ and $LiCa_{1-x}Sr_xAlF_6$ for photonics application. Being doped with Ce^{3+}, Pr^{3+} and Nd^{3+} ions these matrices are promising active media for UV, Visual and IR spectral ranges, correspondingly [1-4]. We report on spectral-kinetic and laser properties on these materials and discuss the dependencies of impurity ions segregation, optical quality and laser characteristics on cationic composition of the crystals.

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6B.3

Synthesis and spectral-kinetic properties of potential down-conversion materials for solar cells based on $Ba_4Y_3F_{17}$, GdF_3 and YF_3 doped with Pr^{3+} and Yb^{3+} ions

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ORAL

Ions pair Pr^{3+}-Yb^{3+} appears to be sufficiently promising for down-conversion to IR spectral range application to improve the silicon solar cells efficiency based on quantum cutting phenomenon[1]. Here we present the investigation of spectral kinetic properties of this pair in $Ba_4Y_3F_{17}$, GdF_3 and YF_3 fluoride matrices, which are characterized by low phonons energy.

$Ba_4Y_3F_{17}$: Yb: Pr samples were synthesized by co-precipitation from aqueous solutions technique from corresponding nitrates and potassium fluoride as a fluorinating agent. GdF_3: Yb: Pr and YF_3: Yb: Pr samples were synthesized at vacuum oven at 1000 ^{0}C from individual fluorides.

Luminescence spectra under excitation at around 445 nm show well-known picture of Pr^{3+} transitions from ^3P_J states together with Yb^{3+} luminescence at around 1000 nm. At the same time, it is known that Pr^{3+} ions may depopulate the $^2F_{5/2}$ state of Yb^{3+} ions through $^1G_4$ state[2] and we see the $^3P_0$-$^1G_4$ luminescence for all samples studied here. In total, all samples of matrices exhibit significant energy transfer to Yb^{3+} ions but $Ba_4Y_3F_{17}$ samples show more intense Yb^{3+} luminescence. The external quantum yield of down-conversion luminescence measured with integrating sphere appeared to be at around 2.5 % for $Ba_4Y_3F_{17}$ matrix, and around 1 % for GdF_3 and YF_3 matrices. Peculiarities of energy transfer and other processes governing the down-conversion efficiency in the investigated compounds are discussed.

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7B.1

INVITED

Nanocrystalline hexaferrites – properties and applications

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Hexagonal ferrites, named due to their hexagonal structure also hexaferrites, are magnetic oxides composed of iron, oxygen and other elements like barium, strontium, or a combination of these. Hexagonal ferrites, including the most popular $BaFe_{12}O_{19}$ barium hexaferrite are based on the low cost components and belong to the most widespread magnetic materials, covering nearly 90% of the global market. They are applied for miscellaneous industries, like permanent magnets for household appliances, loudspeakers, electric motors, magnetic data storage materials, and also as components in various electrical devices. The rapid increase of interest in hexaferrites observed in the last decade, stems however from more sophisticated applications for mobile and wireless communications at microwave GHz frequencies, stealth technologies, and electromagnetic wave absorbers preventing harmful electromagnetic mutual interferences and for health protecting enabling reduction of electromagnetic smog level.

In our talk we present the most promising candidates for abovementioned exotic applications *i.e*. co-doped $SrFe_{12}O_{19}$ (SFO) M-hexaferrites that combine both excellent magnetic and dielectric properties and also theirs nanocomposites of hard magnetic $SrFe_{12}O_{19}$ hexaferrite with soft magnetic $CoFe_2O_4$ spinel mutually coupled by exchange spring interaction. We discuss how co-doping with various elements (like Al, Nd and Sc ions) allows reducing amounts of parasitic phases, modifying the grain morphology and tailoring the properties like electric conductivity, magnetization and coercivity due to effective pinning of magnetic domains. On the other hand the proper processing of the nanocomposites enhances intergranular exchange spring interaction improving their magnetic properties.

We also discuss how structure, grain morphology and magnetic and dielectric properties of these hexaferrite ceramics and theirs nanocomposites influence performance of these materials at microwave frequencies.

7B.2

Selective area formation of GaN nanowires by the use of amorphous Al_xO_y nucleation layer

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We recently reported that self-assembled formation of GaN nanowires (NWs) can be achieved by plasma-assisted MBE on crystalline sapphire if the substrate is covered by an amorphous Al_xO_y (a-Al_xO_y) layer grown by atomic layer deposition [1]. Moreover, much faster nucleation of GaN NWs was observed on such a-Al_xO_y films than on commonly used nitridated Si substrates (Si_xN/Si) under the same growth conditions [2, 3]. Despite these promising advantages, high density of NWs and controlling of their spatial distribution on a substrate still remain an issue.

In this work GaN growth was performed on GaN/sapphire and Si_xN/Si substrates with 12 μm wide stripes of 15 nm thick a-Al_xO_y. Formation of GaN NWs well-aligned perpendicularly to the substrate surface was found on a-Al_xO_y stripes, while a rough compact GaN layer was obtained on bare, crystalline parts of GaN/sapphire template. Since the compact layer forms under N-rich conditions while the growth of GaN NWs takes place under local excess of Ga [4, 5], the ratio of NW length *h* to the thickness of the compact layer *d* can be tailored by adjusting the Φ_N/Φ_{Ga} flux ratio and the growth temperature. The respective modelling was performed taking into account the NW incubation time as a function of growth parameters. In agreement with calculations we found that the value of *h/d* ratio can be increased by increasing the Φ_N/Φ_{Ga} flux ratio. On the other hand pure SAG was observed on Si_xN/Si substrates with a-Al_xO_y stripes. This was achieved by adjusting the growth parameters that nucleation of GaN on Si_xN was prevented (incubation time longer than the growth duration), so GaN NW growth took place on a-Al_xO_y stripes only. Importantly, the same effect of pure SAG was found for PECVD-deposited Si_xN on GaN/sapphire templates. In that way, our results pave the way for SAG of GaN NWs on a wide variety of dissimilar materials by using a-Al_xO_y as a nucleation layer.

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ORAL

7B.3

On the modifications of Pechini method for $Bi_2ZnB_2O_7$ nanoparticles synthesis

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ORAL

Synthesis of $Bi_2ZnB_2O_7$ nanoparticles via modified Pechini method is reported. The introduced modifications are based on proper selection of complexing and polymeric agents and on optimization of process parameters. A polymeric system of EDTA and mannitol was applied. Its thermal behavior is discussed, based on the thermogravimetric measurements [1]. The synthesis temperature and duration (in comparison to conventional route) were reduced. The phase uniformity of obtained powders was studied using X-ray diffraction and crystallographic calculations were done. The morphology of synthesized materials was investigated by means of scanning electron microscopy and atomic force microscopy [1,2]. The influence of organic part quantity on $Bi_2ZnB_2O_7$ particles size and their agglomeration is discussed. The electrophoretic measurements for $Bi_2ZnB_2O_7$ powder were performed to evaluate its isoelectric point. Owing to deagglomeration process and filtration, nanofraction of $Bi_2ZnB_2O_7$ particles was effectively separated.

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7B.4

Scattering-Type Scanning Near-field Optical Microscopy and Spectroscopy for Nanoscale Chemical Analysis

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Scattering-type Scanning Near-field Optical Microscopy (s-SNOM) is an optical microscopy and spectroscopy approach based on scanning probe technology, bypassing the ubiquitous diffraction limit of light to achieve a spatial resolution below 20nanometers. s-SNOM employs the strong confinement of light at the apex of a sharp metallic AFM tip to create a nanoscale optical hot-spot. Analyzing the scattered light from the tip enables the extraction of the optical properties absorption, reflectivity) of the sample directly below the tip and yields nanoscale resolved images and nanoscale spectroscopy (hyperspectral nano-FTIR) information simultaneous to topography. This presentation we will introduce the basic principle of near-field microscopy and hyperspectral nano-FTIR for imaging and spectroscopy with 10 nanometer spatial resolution. In addition we will summarize the latest achievements in the field of near-field microscopy and spectroscopy on polymers, biomaterials and 2D materials and will focus on applications in chemical analysis and material identification at the nanoscale.

ORAL

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9B.1

INVITED

On the mechanisms of (bio)crystal nucleation

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In recent years, the important role of amorphous precursors and intermediates in crystallization has become obvious. While the advantage of such species for the generation of complex crystal morphologies in biomineralisation is known since the early 2000s,[1] the underlying structural, thermodynamic and kinetic characteristics, as well as pathways involved towards the final crystal structures, often remain elusive. Here, we present the newest insights into solute,[2] liquid[3] and solid[4] amorphous precursors and intermediates and their role in crystallization. Thereby, we focus on calcium carbonate ($CaCO_3$), the most abundant biomineral, which is the major inorganic component in mollusk shells, corals or sea urchins —also addressing recent debates.[5] One particular emphasis also concerns the effects of pH levels, which significantly influence $CaCO_3$ precipitation. However, physiological, i.e., near-neutral pH levels have not yet been explored quantitatively. In this study, we implemented a quantitative titration assay to fill this gap. The data shows that although calcium bicarbonate ion association is very weak, bicarbonate binding plays a distinct role during nucleation of the initial mineral phase at near-neutral pH. Solid-state nuclear magnetic resonance (NMR) results evidence that significant amounts of bicarbonate ions are incorporated into the structure of amorphous $CaCO_3$. Indeed, certain recombinant proteins from biomineralization show effects at physiological conditions, which are not evident at higher pH levels, and shed light on the biochemical mechanisms regulating mineral nucleation and growth. Since charge and structure of the proteins do not change in the investigated pH regime, this strongly suggests a role of the bicarbonate incorporation during mineral-protein interactions. Altogether, our work suggests that biomineralization encompasses bidirectional processes involving; (i) biomolecules that modulate nucleation and crystallization behavior of inorganics; and (ii) inorganics that tune the self-association of biomolecules.

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9B.2

Impact of bacteria on crystallization and aggregation of selected components of infectious urinary stones

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One of the types of urinary stones are infectious stones, which constitute from 10 to 30 % of all types of stones diagnosed in humans. The formation of these stones is associated with a urinary tract infection caused by urease-producing bacteria. In 70% of cases these are bacteria *Proteus mirabilis*. Infectious urinary stones consist of an agglomerate of bacteria, highly crystalline struvite, and poorly crystalline and amorphous precipitate (PCaAP). Struvite occurs in the form of crystals with a well-defined habit. PCaAP does not show visible crystalline features and occurs in the form of aggregates without a particular organization[1]. Carbonate apatite, $Ca_{10}(PO_4)_6CO_3$, (CA) and hydroxylapatite, $Ca_{10}(PO_4)_6(OH)_2$, (HAP) may be classified as the poorly crystalline phases[1]. CA and HAP may exist in non-stoichiometric forms. The amorphous phases that are components of infectious urinary stones include: amorphous calcium carbonate (ACC), amorphous calcium phosphate (ACP), and/or amorphous carbonated calcium phosphate (ACCP)[1]. The infectious urinary stone becomes large not because of single crystal growth, but because of the agglomeration of large amount of relatively small crystals (tens of micrometers) of struvite with small deposits of PCaAP and bacteria. Therefore, the aggregation is supposed to be one of the primary causes of urinary stone formation. From the three components: struvite, PCaAP and bacteria, PCaAP has the greatest ability to aggregate[2]. The present study describes an experimental results[3] on the aggregation of PCaAP in the presence and absence of *Proteus mirabilis*. The aim of the present study is also to analyze the aggregation of PCaAP in the presence of bacterial macromolecules obtained from *Proteus mirabilis* culture with lipopolysaccharide (LPS) and without it. Optical microscopy methods and zeta potential measurements both confirm that the bacterial LPS is the key factor promoting aggregation[3].

ORAL

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9B.3

ORAL

An insight into coarse-grained protein models in the context of applying them to protein crystallization

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In many cases, the process of crystal growth is preceded by a stage of theoretical modelling, which in turn is supported by computer simulations. Computer models can be divided into full-atom and coarse-grained models. Due to the complexity of the modelled systems, full atomic simulations are very time-consuming. Already in the seventies of the twentieth century, the first attempts were made to simulate protein folding using reduced models[1]. In the following years, many coarse-grained models[2] were developed in which the level of graining ranged from small groups of atoms[3] (e.g. few heavy atoms and associated hydrogens) or molecules[3] (e.g. water) to the whole proteins[4]. In the case of the latter, patchy models[5] stand out. Depending on the real shape of the biomolecules and the degree of generalization, their model counterpart may be spherical, ellipsoidal or in the form of a baton and even cubes[6]. Their physicochemical properties are determined by uniformly, accidentally or specifically distributed patches on the surface of the model molecule[7].

The main groups of coarse-grained models with their advantages and limitations in terms of their applicability to protein crystal growth modelling will be presented and discussed.

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10B.1

Homogenization and Decomposition of InGaN Quantum Wells at Elevated Temperatures due to Indium Atoms and Point Defects Diffusion

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The subject of our interest are the studies of an influence of p-type layers growth temperature on changes of indium distribution within the InGaN/GaN Quantum Wells (QWS) just before their decomposition and the decomposition process itself.

To investigate how the growth temperature of p-type layers affects QWs emitting light in blue-cyan region, we used LED structures with QWs overgrown by p-type layers in temperatures range 830ºC – 930ºC. Electroluminescence (EL) data show us that just before decomposition at 930ºC, optical power of the emitted spectra increase, and at the same time, wavelength and FWHM decrease with increasing p-type growth temperature up to 905ºC. These results strongly suggest that just before decomposition of the QWs, In atoms homogenization process take place. HR-XRD measurements show that these changes are related only to decreasing average indium content in MQWs without worsening of the structure what is in good agreement with EL measurements. TEM images confirm also that decomposition of MQWs up to 905ºC in case of blue-cyan emission does not occur. When we are close or just in the moment of the decomposition of the MQWs (in this case 930ºC) we can observe in HR-XRD measurements, worsening of the quality of MQWs, oscillation (Pendelosung fringes) almost disappear. Wavelength and FWHM of measured EL peaks dramatically increase and optical power decrease.

One possible explanation of the observed changes in EL and HR-XRD characteristics is the reduction or rearrangement of the indium clusters within InGaN MQWs before their decomposition.

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INVITED

10B.2

ORAL

Microspheres for WGM resonators for use as biosensors

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Whispering gallery mode (WGM) microresonators have variety of applications, such as optical switches, narrow band lasers or thermo- and biosensors.[1,2]

We develop microresonators from fused silica and also from low-melting materials (like tellurite and phosphate glasses), which were not previously used for this purpose. Our group developed for the first time the resonator, based on Na5[B2P3O13] (NBP) phosphate glass doped with both quantum dots and plasmonic nanoparticles using the NanoParticle Direct Doping method[3], which is based on one of the crystal growth techniques - the micro-pulling down method.

WGM microresonators are excited, by a special system that allows measurement of WGM resonances. In our system we use tapered fiber coupling for exciting WGM microresonators.

The aim of our work is to utilize the microresonators for detection of exosomes, which are small vesicles excreted by every cell in our body to body fluids. By detecting exosomes from cancerous cells we want to perform fast, early and non-invasive cancer diagnostics.

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Device Quality GaSb Epilayers Grown on Si (001) Substrates

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GaSb is a group III-V compound which is very crucial for optoelectronic devices not only as an active epilayer but as an important substrate. Although it is an advantage to use GaSb as a substrate for Sb-based devices due to its lattice match structure, the production costs of GaSb substrates are relatively high compared to GaAs and Si ones. Owing to the fact that it is a very important substrate, an alternative approach for the replacement of GaSb substrate is a highly attractive research interest considering lowering the production cost of Sb-based devices [1]. In this study, device quality GaSb epilayers grown by molecular beam epitaxy on Si substrates are presented as an alternative to GaSb substrates.

GaSb films were grown on Si (001) by molecular beam epitaxy via III-V quantum dots as an interfacial misfit (IMF) array between Si substrates and GaSb films. The effect of IMF array thickness, growth temperature and post annealing on the quality of GaSb films grown on Si were monitored by high resolution X-ray diffraction, photoluminescence, scanning electron microscopy (SEM) and atomic force microscopy (AFM). An RMS value of ~1 nm for a scanning area of 100 μm^2 was obtained from the sample containing 20 ML IMF thickness, grown at 530 ^oC and post annealed at 550 ^oC for 30 min. The obtained RMS value is quite important for growing Sb-based devices on Si substrates [2].

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S.1

ORAL

Automated evaluation of 2D Diffraction Data

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A lot of research effort is spent on the development of band gap engineered products such as HEMTs or LEDs. These typically contain layered structures of increasing complexity. X-ray diffraction is one of the basic analytical methods that is routinely utilized for both materials research and quality control in production. In addition to the non-destructive nature of X-ray scattering methods, it delivers very detailed structural information on epitaxial layers and substrates including strain, strain relief, film thicknesses, crystalline quality and mosaic spread.

Recent advances in area detector technology (GaliPIX^{3D} and PIXcel^{3D}) combined with smart positioning algorithms and signal processing allow now reciprocal space maps (RSM) to be recorded in similar timescales as rocking curves. These high-speed measurements find applications in the characterization in all crystalline advanced materials. Position sensitive detectors allow to collect many points at once and can be used in continuous mode, so less measurement and positioning steps are necessary to build up a given area of reciprocal space. This contribution will discuss opportunities and limitations of this technique in conjunction with automatic evaluation of such data to create xy wafer maps that display the buffer layer relaxation state. The fully automated workflow enables new possibilities of X-ray metrology for mismatched layer stacks.

Advantages for the growth community:

- Very fast feedback about growth results regarding layer relaxation and ternary compositions within minutes
- Preview ultra-fast reciprocal space maps before starting detailed measurements
- Crystal analyzer for high-resolution mode and ultra-fast RSM setup work complimentary on one XRD platform

Keywords—High-resolution XRD; RSM; epitaxy;

Raman spectroscopy application in materials science

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Raman spectroscopy (and Raman imaging) has become a powerful, noninvasive method to characterize a wide range of materials including 2D matrials. A large amount of information such as disorder, edge and grain boundaries, thickness, doping, strain and thermal conductivity of graphene and other 2D materials can be learned from the Raman spectrum and its behavior under varying physical conditions

In this work we compare and contrast the different solutions for maintaining focus and conducting Raman imaging on 2D materials with uneven, complex surfaces. We describe and illustrate the application of the new LiveTrack™ dynamic focus tracking technology, which not only provides in-focus Raman images of the most challenging samples but also topographic information, allowing three dimensional surface Raman images to be generated.

We discuss and present data on a range of extremely difficult samples including graphene on a Cu foil, a sample that is rough on a micrometre length scale. We will demonstrate dynamic measurement of a polyethylene pellet undergoing phase transitions in a temperature cell, demonstrating that LiveTrack can be used to maintain focus in moving systems.

As a complete picture of novel 2D materials characterization (like ReS_2) a new tool of low frequency Raman imaging will be shown, including share modes and breathing modes analysis.

Keywords: Raman spectroscopy, 2D materials, imaging

Posters

Czochralski growth of Phosphorus and Boron doped Germanium

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For the Czochralski (Cz) growth of P- and B-doped Ge, the development of a new doping method from the gas phase is required. The problem with phosphorus doping ($k_0 = 0.08$) of Ge is due to the lack of stable dopant sources. Therefore, the gaseous chemical compound phosphine (PH_3) was chosen as a phosphorus source. P-doped Ge crystals were grown in a Floating Zone (FZ) furnace using the so-called Mini-Cz method.[1] A peculiarity of this method is the use of inductive heating with a frequency of f≈3MHz. This allows stable growth conditions, even in the case of very small quantity of starting material. The Ge crystals were grown using quartz crucible with 1 kg polycrystalline Ge charge. Doping was carried on by blowing PH_3 to the melt surface using a special nozzle. When the dopant gas reaches the melt surface, having at least the melting temperature of Ge (938°C), it disintegrates 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 then the growth starts. In this manner, <100> P-doped Ge crystals up to 50 mm in diameter with a charge carrier density in the range from 1×10^{14} cm^{-3} to 5×10^{17} cm^{-3} were grown.

The same doping approach was also used for B-doped Ge crystal growth. As a boron source, the gaseous chemical compound diborane (B_2H_6), normally used for B-doping during FZ growth of silicon, was chosen here. Since the distribution coefficient of B in Ge is $k_0 > 1$ (k_0 is equal to the value between 5 and 22 according to different sources) [2], the Ge melt will lose Boron during the growth and its concentration in the growing crystal will eventually drop. This necessitates the replenishment of B losses by continuously blowing diborane to the melt surface. Further, distance between the gas nozzle and the melt surface was held near constant by crucible translation thereby compensating the melt level-lowering. Charge carrier density in the range between 1.5×10^{14} cm^{-3} and 2.5×10^{16} cm^{-3} was achieved in four <100> B-doped Ge crystals grown.

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Influence of growth conditions on defect types and densities in 4H-SiC

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Silicon carbide (SiC) is a semiconductor with a high application variety but often a poor crystal quality, which negatively influences the device's performance [1]. SiC has a high diversity of defects, like Micropipes (MP), Threading Screw Dislocation (TSD), Threading Edge Dislocation (TED), Basal Plane Dislocations (BPD) and so on [2]. Several SiC wafers, grown by Physical Vapor Transport [3] at various growth conditions were characterized with different X-ray methods. Laue precision measurements were carried out to obtain angle and direction of misorientation. Synchrotron White Beam X-ray Topography (SWXRT) in back-reflection geometry was used to determine defect types. High resolution X-ray diffractometry (HRXRD) measurements were performed at the same positions to investigate the crystal quality in terms of crystal lattice strain and tilt. SWXRT were performed at the TopoTomo Beamline, IPS Karlsruhe institute for technology (KIT). The dominating defect types include TSDs, TEDs, MPs in different densities. Between screw dislocations small-angle grain boundaries are located. Additionally, full wafer mappings in transmission geometry show a difference in defect density depending on the growth conditions. The wafers grown at lower temperatures, with a higher growth duration, a lower growth rate and a higher crystal diameter indicated a lower defect density and a limitation of the defects to the wafer's edge. Furthermore, the mappings showed a clear lineation of small-angle grain boundaries in direction of the wafer's misorientation. Therefore appearance and development of small-angle grain boundaries are in direct relation with angle and direction of the crystal's misorientation. With the HRXRD Rocking curves were recorded in 0004 reflection showing a difference in their curve shape and full-width at half maximum (FWHM), depending on the crystal quality. Reciprocal Space maps were recorded at the same positions in the same reflection. Depending on the dominant defect type in the measured area the relation of crystal lattice strain to crystal lattice tilt changes. A higher density of TSDs and small-angle grain boundaries induces more crystal lattice tilt. The results of all measurements showed a high defect density in the wafer but inhomogeneous distribution, pointing to an inhomogeneous temperature distribution and a high temperature gradient vertically and horizontally during growth.

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P.3

Antisolvent crystallization of aqueous ammonium dihydrogen phosphate solutions containing of Fe(III) impurity by addition of ethanol

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POSTER

Experimental results of antisolvent crystallization of ammonium dihydrogen phosphate from its aqueous solutions saturated at 30 °C containing different concentrations c_i of Fe(III) impurity by addition of ethanol at preselected feeding rate R_A, studied by *in situ* measurements of solution temperature T as a function of feeding time t of ethanol at rate R_A, are discussed. The effect of addition of the antisolvent on aqueous ADP solutions containing different concentrations c_i of Fe(III) impurity during crystallization was compared with that on pure water where the Fe(III) ions do not change plots of T against t during the addition of ethanol. As in the case of pure ADP solutions without impurity, the plots of ΔT against t in the presence of different concentrations c_i of the Fe(III) impurity reveal three well-defined regions: (1) nucleation associated with endothermic reactions, (2) crystal growth associated with exothermic reactions, and (3) solution cooling associated with supersaturation decay with increasing time (partly region of endothermic reactions). Region (1) of ethanol feeding duration between t_0 and t_1 where $\Delta T < 0$ can be used to investigate MSZW of ADP solutions[1,2]. It is found that the value of MSZW, as determined by the value of the maximum antisolvent content Δx_{max}, for a given R_A increases with increasing impurity concentration in saturated ADP solution.

The experimental $\Delta x_{max}(R_A)$ data for different concentrations c_i of Fe(III) impurity are analyzed by using two dependences: $\ln(\Delta x_{max}) = \Phi + (1/m)\ln R_A$ and $(\Delta x_{max})^{-2} = F(1 - Z\ln R_A)$, with constants Φ, m, Z and F, predicted by self-consistent Nývlt-like approach and an approach based on the classical three-dimensional nucleation theory, respectively[1,2]. The results are also compared with the parameters obtained for the system without impurities.

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P.4

POSTER

Theoretical and experimental determination of selected properties of the struvite crystal

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Magnesium ammonium phosphate hexahydrate, $MgNH_4PO_4 \cdot 6H_2O$, known as struvite, remains in focus of research in physical chemistry for a number of reasons. First of all, precipitation of struvite may lead to clogging wastewater system pipes. As a material rich with phosphorous, nitrogen, and magnesium struvite is a main compound recovered from wastewater and recycled as a useful P-N-Mg-containing fertilizer. Recently, a growing interest in investigating properties of struvite has been ignited by the fact that it constitutes the main component of the so-called infectious urinary stones.

Better understanding of the struvite crystal growth and accurate determination of its physical properties is therefore of crucial importance. Unfortunately, experimental investigations have been rather scarce and not systematically followed by theoretical investigations. Recent advent of modern density functional theory quantum chemistry tools makes it now feasible to predict theoretically properties of struvite and exploit them in interpreting or validating experimental data.

We have recently measured highly accurate lattice constants for struvite and confronted them with the theoretically obtained ones. It has enabled us to narrow down a number of density functionals to a few, best performing, ones. Special care has been paid to investigating the role of the used basis sets and the basis set superposition error. The established computational methodology has been subsequently applied to compute vibrational spectrum of struvite. A good agreement between experimentally and theoretically obtained spectra has enabled for theoretically-aided analysis of the IR spectrum. On the other hand, the measured UV-Vis spectrum of struvite, leading to estimation of the optical gap, has confirmed that density functionals lead to predicting energy gaps of dielectric materials in large and unsystematic errors.

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P.5

Growth and Characterisation of Triglycine Sulfate under the Influence of Surfactant Additives

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POSTER

This project investigates the effect of surfactant additives on growth kinetics, morphology, and electric properties of Triglycine sulfate (TGS) crystals. As the pyroelectric Figure of Merit (FOM) is largest in [010] direction, stabilising the (010) face would allow for larger wafers, if FOMs are not heavily impacted. For this purpose TGS crystals were grown from pure TGS solution, and those with 0.388 mol% of Sodium dodecyl sulfate (SDS) and Sodium 1-decanesulfonate (S1-DS), respectiveley, employing a temperature lowering method. While S1-DS TGS crystals show elongation along [010], SDS TGS crystals exhibit larger (010) faces. For both samples the UV-visible transmittance is reduced, compared to pure TGS. At the same time the relative permittivity of the samples at room temperature is decreased from 105 to 75 for SDS TGS, and to 55 for S1-DS TGS. The pyroelectric coefficients were determined using a dynamic measuring method[2]. All samples show the same pyroelectric coefficient of 168 μC/(m^2·K) at room temperature. Thus the decreased permittivities cause voltage generation FOMs to increase proportionally from 201 kV/(m·K) for pure TGS to 245 kV/(m·K) for SDS TGS and 317 kV/(m·K) S1-DS TGS respectively[1].

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P.6

POSTER

Float-Zone growth of silicon crystals using large area- seeding

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The large-area seeding concept was applied for Float-Zone (FZ) growth of monocrystalline silicon, without the common Dash-technique [1] for elimination of dislocations. Using large-seeding avoids the need for growth of a crystal cone with a smaller diameter than the wafer size. Crystals with 4 inch diameter and 120 mm length were grown using a modified FZ set-up [2]. This is characterized by avoiding a thermal shock resulting in dislocation generation during seeding and reduced thermal stress during heat-up and the subsequent crystal growth process. Characterization results showed that the grown material was partly monocrystalline but not dislocation-free.

The main goal of the numerical simulations was to minimize thermal stress down to levels that allow monocrystalline growth. The thermal stress field in the crystal during growth was simulated using a developed numerical model of the process [3]. The calculated von Mises stress was by more than 50% lower than during classical dislocation-free FZ growth, but still by more than a factor of 10 higher than the critical shear stress for dislocation multiplication.

It was concluded that dislocations from the seed surface were mobilized during heat up and introduced to the bulk of the seed, before the actual start of the crystallization. The simulations showed that an extension of the monocrystalline domain was possible to some extent, which was verified by the experimental findings. Further improvement towards lower stress levels requires a different set-up that is less similar to the classical FZ method, which inherently involves high thermal stress.

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P.7

Growth rate and crystalline quality of PVT grown AlN crystals depending on temperature and axial gradient

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AlN bulk crystals, grown by physical vapor transport (PVT) at temperatures exceeding 2000°C, are used as substrates for optoelectronic devices such as deep UV LEDs [1]. To grow AlN crystals with low defect densities the relationship between the temperature field (supersaturation) and the growth rate must be considered in order to influence the defect formation.

In this study, we calculate the temperature at the seed (T_{seed}) as well as the temperature difference between source (max) and seed (ΔT) from pyrometer measurements on the crucible top and bottom by numerical modelling (Virtual Reactor for AlN 7.8, STR group). Conducting growth experiments under different growth conditions and correcting for hot-zone degradation issues we could establish a process window in which the growth rate can be reliably predicted using only the values for T_{seed} and ΔT (Fig. 1). Likewise, from these values the required pyrometer temperatures and growth conditions can be determined.

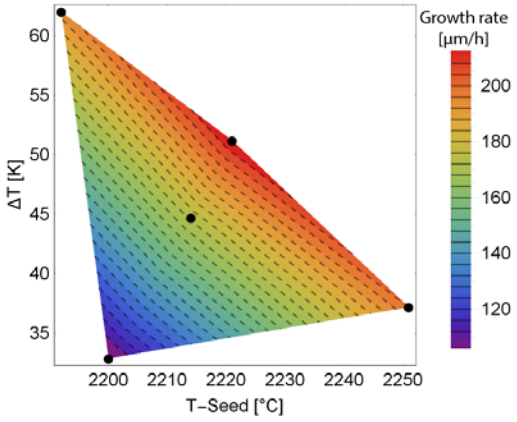

Fig. 1: Calculated AlN PVT growth rate dependence on T_G and dT with experimental data points (black circles)

The obtained data shows a good coincidence with the growth rate model for PVT AlN established by V. Noveski et al. [2]. Additionally, the dislocation density of the grown crystals was determined by defect selective etching and correlated to growth conditions.

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POSTER

P.8

POSTER

Crystal lattice defect structure of selected perovskite-like single crystals of the general chemical formula of $ABCO_4$

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Oxide materials of the general composition $ABCO_4$ (where A = Ca, Sr, Ba, B = La, Nd, Pr, and C = Al, Ga) with layered perovskite-like structure are of interest due to their possible applications as substrate materials for high-temperature superconductors, host materials for laser media and phosphors as well as elements of other optical and electronic devices [1 – 3]. Crystal lattice defects could limit considerably possible applications of the crystals. Thus, understanding of mechanisms of the formation of crystal lattice defects is important from technological point of view.

The aim of the work was to study the real structure of $ABCO_4$ single crystals, such as $CaNdAlO_4$-$SrNdAlO_4$ (CNA-SNA) solid solutions and $SrLaGaO_4$ (SLG) single crystals. The analysis of the defect structure was performed in relation to selected growth conditions and stoichiometry. Investigations were performed by means of various methods, especially by X-ray diffraction topography, which enables one to detect and characterize crystal lattice defects formed during the growth process. Selected elements of the defect structure, such as the cellular structure in the outer part of the crystal and rows of rod-like volume defects in the core of the SLG single crystals grown in the [001] direction, were characterised in detail. As a result of the analysis of diffraction contrast two simplified models were proposed: one for the formation of the diffraction contrast associated with rod-like volume defects in the core of SLG crystals and the other one related to the crystal lattice deformation around these defects.

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P.9

The influence of sodium dodecyl sulfate on the growth and morphology of Triglycine sulfate

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POSTER

Triglycine sulfate (TGS) is used for applications as high sensitivity infrared detector, but also for sensor devices due to its strong piezo- and pyroelectric effects, which are the highest along the b-axis. The [010] directions of pure TGS has the highest growth velocity, therefore the (010) faces disappear during growth and a corner or edge will appear in the [010] direction[1]. To slow down the growth velocity in [010] direction and to stabilize the (010) face, detergents are used. J. Fammels[2] worked with different additives and sodium dodecyl sulfate (SDS) slowed the growth in [010] direction significantly. Therefore further growth runs are performed with 0.0338 Mol% SDS and 0.00338 Mol% SDS, using a temperature decreasing technique. The growth results will be presented with special respect to the growth morphology. The orientations of the crystal faces are measured optically and by the Laue method. The crystals grown with 0.0038 Mol% SDS show twins, which are rotated by 180 but as expected the growth velocity in [010] direction decreased and a larger (010) face resulted. Furthermore X-ray powder diffraction and measurements of the dielectric parameters will be presented, showing no significant influence of SDS on the crystal structure and the electrical properties, except the pyroelectric coefficients, which slightly increased. Details about the dynamic method for the measurements of the pyroelectric properties of TGS[3] will be given.

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P.10

POSTER

Recent developments in silicon crystal growth from a granulate crucible

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Electronic applications of silicon are tremendously increasing during the last years. Crystal quality and yield are major issues and are driving the development to improve existing processes or develop new crystal growth methods. For silicon growth Float Zone (FZ) is a high purity alternative to crystals grown by the Czochralski (Cz) process. Both methods for Si crystal growth have crucial disadvantages with regard to quality or cost, respectively[1]. Therefore, a new concept was developed with the potential to avoid the disadvantages and combine the advantages of the established growth methods. This method is called Silicon Granulate Crucible (Si-GC) method based on a Patent[2]. The present goal is to demonstrate a growth process for crystals with a preindustrial relevant diameter of 100 mm. In order to compare these crystals to Cz and Fz material characteristic properties of Si-GC grown crystals have been evaluated.

During the Si-GC growth process, the crystal is pulled upwards out of the melt pool through the hole of a flat inductive coil, as the primary heat source. Continuous replenishment of the feed material (electronic grade Si fluidized bed granulate) is done through a second hole in the coil. Inductive heating instead of graphite heaters reduces carbon contamination in the crystal. On the other hand, the granular bed forms an inherent crucible for the melt[1,2]. Reducing oxygen contamination in the crystal impurity analysis showed low oxygen content ($<1\times10^{16}$ cm^{-3}) on the level of FZ-Si.

Currently, a stable growth process has been developed with monocrystalline structure up to crystal diameter of 60 mm. Axially homogeneous n-type resistivity distribution by using doping with phosphine gas was achieved.

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P.11

DFT studies on the point defects migration in the bulk GaN, InN, AlN and at the InN/GaN and InGaN/GaN interfaces

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POSTER

The nitride semiconductors GaN, InN, and AlN are very attractive for a wide application in optoelectronics. Modern light-emitting devices utilize nitrides as the multi-quantum well structures grown by MOVPE or MBE methods. Efficiency of the light emitting diodes is still a big technological issue especially in green region of spectrum. This efficiency is directly related to the diffusion of point defects in the active parts of devices during its growth and as consequence decomposition of quantum wells at high temperatures during p-type layers growth.

In this work, we present the theoretical calculations based on density functional theory (DFT) concerning the formation energies of defects and potential energy barriers for diffusion of these defects. Initially, we studied single vacancies V_N, V_{Ga}, V_{In}, V_{Al}, double vacancies $2V_N$, $2V_{Al}$, as well as complexes of a substitution atom and vacancy $In_{Ga}+V_{Ga}$, $Ga_{In}+V_{In}$, $Ga_{Al}+V_{Al}$, $In_{Al}+V_{Al}$ in the bulk GaN, InN, and AlN crystals. Additionally, we investigated various charge states of these defects, which play a role of potential donors or acceptors. By means of the Nudged Elastic Band (NEB) method we estimated heights of diffusion energy barriers that are in the range of 0.74 – 5.50 eV for the bulk materials. Next, it was found that for the InN/GaN structure strained to the GaN, the formation energy of nitrogen vacancies are the lowest in the InN layers close to interface. We also discussed several cases regarding diffusion of Ga and In atoms across interface in the presence of additional metal vacancies. The obtained formation energies and diffusion barriers confirm the possibility of effective exchange of these atoms.

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POSTER

Raman spectroscopy of Ce^{3+} doped $LuAlO_3$ and $Lu_3Al_5O_{12}$ single crystalline films

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The perovskite- and garnet-based materials are widely studied for a few decades due to their great technological relevance, where their use as a host materials for rear earth elements gives widespread areas of applications. New perspective for perovskite and garnet materials appeared along with crystallization of single crystalline films (SCFs) by liquid phase epitaxy method (LPE). One of the most exploited and promising field of applications of SCFs of perovskites and garnets is scintillation technology [1].

During the last decade single crystalline films (SCFs) of Ce^{3+} doped $YAlO_3$ and $LuAlO_3$ perovskites [2,3] and $Y_3Al_5O_{12}$ and $Lu_3Al_5O_{12}$ garnets [4,5] were synthesized using liquid-phase epitaxy method.

We present Raman spectroscopy investigation of Ce^{3+} doped $LuAlO_3$ (LuAP) and $Lu_3Al_5O_{12}$ (LuAG) single crystalline films grown onto YAP and YAG substrate, respectively. The Raman spectra and Raman map recorded for LuAP:Ce/YAP epitaxial structures allow a distinction between SCF and the substrate. In the case of LuAG:Ce/YAG SCF in the range of observed vibrational modes additional signals attributed to 4f-4f electronic transitions of rare earth impurities are observed.

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P.13

Growth, spectroscopic and laser properties of heavily doped $LiCaAlF_6:Ce^{3+}$ and $LiSr_xCa_{1-x}AlF_6:Ce^{3+}$ UV active media

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$LiCaAlF_6:Ce^{3+}$ (Ce:LiCAF) crystal is a known and promising active medium for the ultraviolet spectral range [1,2] mainly due to large value of band gap (100 000 cm^{-1}) and significant vibrational broadening of energy levels of Ce^{3+} ions providing advantageous localization of energy states of impurities perspective for UV and VUV light amplification. This lies in the course of industrial trends on shortening the wavelength and meets demand on robust and cheap in use laser sources.

The purpose of this work is to study spectroscopic properties of samples of $LiSr_xCa_{1-x}AlF_6:Ce$ grown by the Bridgman method at various temperatures using different excitation wavelengths and laser properties of the crystals.

As a result of the investigation, three types of impurity centers of Ce^{3+} ions were identified and increase (about 6 times) of Ce^{3+} segregation coefficient in mixed crystals was observed which is in favor of certain types of centers. Comparing the site-selective luminescence studies at different temperatures energy transfer peculiarities were investigated.

Also here we report on laser oscillation achieved from investigated Ce:LiCAF crystals with 1.2 at.% Ce^{3+} content with the threshold near 3.5 mJ and slope efficiency up to 47% at 287 nm which is also high and speaks for high optical quality of the crystals.

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POSTER

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POSTER

Investigation of dislocation networks in high-purity germanium for detector applications

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Recently, there is a growing interest in large high-purity germanium (HPGe) detectors, since they play a key role in answering important fundamental questions in particle physics and cosmology, such as the existence of the neutrinoless double beta decay (LEGEND experiment) and the nature of dark matter.[1,2] For these applications Ge with a high-purity, as well as with a specially tailored defect structure is required. In particular, the dislocation network of the crystal influences vacancy clustering, forms acceptor states, and is associated with charge trapping centers. Desirable for detector use is a uniform dislocation density, controlled within a range of 500 to 5000 cm^{-2} throughout the crystal.[3,4]

The starting material of 6N Ge was gradually purified, first in graphite boats and afterwards in fused quartz boats, in H_2 atmosphere within a specifically constructed zone refiner. For the purpose of process development, two <100> single crystals with a diameter of 2'' were grown by the Czochralski (Cz) process under H_2 and Ar gas flow, respectively. It is well known that HPGe growth needs a dedicated Cz furnace operated under pure H_2 gas flow, due to the requirement of high electrical purity.[3] However, the high thermal conductivity of H_2 compared to Ar makes it particularly difficult to have the right thermal conditions to obtain a low and uniform dislocation density. Therefore, the influence of the used gas on the crystal morphologies and the resulting dislocation distributions within {100} wafers throughout these crystals, is analyzed here. Dislocations were identified by observation of etch pits originating from defect-selective wet-chemical etching. The dislocation distribution of the crystal grown in Ar was investigated in intersecting {100} and {110} longitudinal sections of the crystal shaft, as well as {100} wafers intersecting the longitudinal sections. Clusters of dislocations winding through the crystal and a dislocation density in the order of 10^4 cm^{-2} was observed.

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$Li_2W_{1-x}Mo_xO_4$ crystals grown by low-thermal-gradient Czochralski technique

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Li_2WO_4 crystal is known to exhibit spinel→phenacite polymorph phase transition below melting temperature, which leads to cracking of the crystal after growth process.[1,2] Li_2MoO_4 crystal structure is similar to phenacite-type Li_2WO_4 crystal structure. It is known that polymorph transition can be suppressed by partial substitution of tungsten with molybdenum.

In present work crystals of variable compositions $Li_2W_{1-x}Mo_xO_4$ (x = 0.15, 0.1, 0.05, 0.025, 0.0125) were grown by low-thermal-gradient Czochralski technique (LTG Cz). Characteristic feature of LTG Cz are temperature gradients below 1 deg/cm that prevents overheating of the melt and volatilization of melt components and reduces thermoelastic stress in growing crystals.[2] Initial materials were synthesized from extra-pure Li_2CO_3 (Novosibirsk Rare Metals Plant, TS 6-09-4757-79) and deeply purified MoO_3 and WO_3.[3]

$Li_2W_{1-x}Mo_xO_4$ crystals with 2.5 and 1.25 mol.% of molybdenum were grown for the first time. Crystals with 15 mol.% of molybdenum had large amount of defects, crystals with less molybdenum ratio had low amount of defects and colourless that characterizes them as crystals of high optical quality. Test samples were cut out from obtained crystals for further study.

Fig. 1. Typical form of $Li_2W_{1-x}Mo_xO_4$ crystal, x = 0.1

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P.16

POSTER

Morphology and optical properties of precipitated vivianite, $Fe_3(PO_4)_2 \cdot 8H_2O$

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Crystals of vivianite, belonging to the monoclinic prismatic class 2/*m*, have been grown by precipitation from aqueous solution at temperatures from 25 to 56°C. The crystals obtained are tabular along {010}, and further forms observed are {110} and {-101}, in a few cases {001} as well.

At 25°C aggregates were dominating, but from 35°C and above single crystals were more common; in a few cases twins were observed. The crystals were, however, in general rather irregular. At low temperature the outline was distinct and consistent with the above indicatiopns and published lattice parameters,[1] but the {101} form was hardly visible, and the {010} faces were very rough as judged from interference colours seen in the polarising microscope.

Lateral faces belonging to the {110} form are visible on crystals grown at 45 and 56°C. The width of the projection of such a face on {010} may be measured with an ocular micrometer and thus serve for estimating crystal thickness, making use of the face angle calculated from lattice parameters.

Optical parameters were determined by a combination of three methods: 1) measurement of birefringence with an Ehringhaus 6λ compensator, making use of crystal thickness determined as indicated above, 2) visual interferometry using a polarising microscope incorporating a Mach-Zehnder interference device (Zeiss Jenapol Interphako),[2] and 3) Kamb's method for the optical axes angle determined by the angle-of-flight method in conoscopic illumination.[3] Resonably precise values of principal refractive indices in the expected range, according to literature, were found.

Some of the crystals in a sample prepared 6 years ago, mounted in Canada balsam on glass slides and stored in a black box since then, had turned blue, whereas other crystals in the sample were still colourless. The blue crystals showed strong pleochroism.

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Research of microcrystal Bi_3TeBO_9 doped with rare-earth ions by Raman spectroscopy

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Recently, there has been an interest in bi-functional materials that combine nonlinear optical properties as well as luminescence properties in one system.

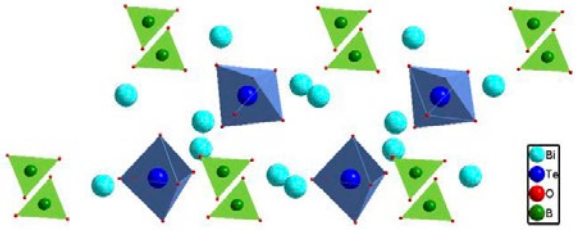

Fig.1. The structure of Bi_3TeBO_9

Investigated Bi_3TeBO_9 microcrystalline powders doped with active RE^{3+} ions are characterize by the high efficiency of the second harmonic generation of matrix and exhibit effective luminescence of active ions [1,2]. The powders were synthesized by means of the modified Pechini method [3]. In this work, we present the results of investigations of vibrational properties Bi_3TeBO_9:Nd^{3+}powders studied by Raman spectroscopy. Moreover, the characteristic emission of Nd^{3+} ions at about 890 and 1062 nm ($^4F_{3/2} \rightarrow {}^4I_{9/2}$ and $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transitions, respectively) were measured and analysed.

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P.18

Spectroscopic and nonlinear optical properties of $Bi_2ZnOB_2O_6:Ln^{3+}$ (Ln^{3+}: Nd^{3+}, Er^{3+}, Yb^{3+}/Er^{3+}) crystalline materials

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POSTER

Among various crystalline materials doped with trivalent rare earth ions, those doped with the neodymium (Nd^{3+}) and erbium (Er^{3+}) have attracted a great interest and are constantly and extensively studied due to their importance for optical communication,[1] laser technology[2] and for development of the new optoelectronic devices.[3] Materials doped with Er^{3+} ions usually exhibit emission around 1535 nm ($^4I_{13/2} \rightarrow {}^4I_{15/2}$ transition), which can be enhanced by co-doping with ytterbium (Yb^{3+}) ion, while those doped with Nd^{3+} show emission around 1060 nm ($^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition). Reported by us, a new $Bi_2ZnOB_2O_6$ crystalline materials doped with Nd^{3+} and Er^{3+} or Yb^{3+}/Er^{3+} seem to be promising materials with numerous potential applications. In the present work spectroscopic properties of $Bi_2ZnOB_2O_6:Ln^{3+}$ (Ln^{3+}: Nd^{3+}, Er^{3+}, Yb^{3+}/Er^{3+}) single crystals and crystalline powders are presented. $Bi_2ZnOB_2O_6:Ln^{3+}$ single crystals were grown from stoichiometric melts by means of the Kyropoulos method, while crystalline powders were obtained by means of a modified Pechini method. Moreover, the nonlinear optical properties of $Bi_2ZnOB_2O_6:Ln^{3+}$ single crystals are reported. Finally, due to high values of nonlinear optical coefficients as well as the effective luminescence of excited ions, $Bi_2ZnOB_2O_6:Ln^{3+}$ in the form of single crystals and crystalline powders can be very useful for the integrated optical devices.

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P.19

Semiconducting eutectic composites for photoelectrochemical water spitting

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Increasing energy consumption requires new energy sources. Due to high energy conversion efficiency and carbon free emission, hydrogen becomes an important alternative fuel. One of the ways of hydrogen generation is photoelectrochemical (PEC) water splitting into oxygen and hydrogen under the solar energy.

Current research on PEC cell focuses on improving its performance by designing composite cells consisting different semiconductors. Eutectic composites are a good alternative materials for solar-energy conversion due to: (i) high crystallinity, (ii) sharp interfaces between phases, (iii) wide variety of possible composites, and (iv) ability to modify properties by: doping, annealing and/or etching.

Laboratory of Functional Materials of Institute of Electronic Materials Technology fabricates and widely studies eutectic systems in example of different geometrical motifs,[1,2] plasmonic effect,[3] and others. The authors will present the actual research on eutectic systems fabricated by the micro-pulling down method for the application in PEC, including microstructural and photoelectrochemical characterization.[4,5]

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P.20

POSTER

Czochralski growth of large (dia. > 60 mm) YAP:Ce crystals and its improved scintillation properties

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Yttrium aluminium perovskite doped with cerium ($YAlO_3$:Ce, YAP:Ce) is a fast, mechanically and chemically resistant scintillating material. Single crystal YAP scintillators have many interesting features for gamma, X-ray and electron detection[1], e.g. short decay time of 25 ns, good energy resolution, negligible afterglow and sufficient light yield even at elevated temperatures of 200 °C.

YAP:Ce emission typically overlaps with tail of broad absorption (due to color centres) causing partial reabsorption of emitted radiation and decreasing the effective light output. This limits the application for volume detectors as light is attenuated along the length of the crystal. The self-absorption can be evaluated as absorption length[2].

We report successful Czochralski growth from YAP:Ce melt; the size of the pulled crystals is above 60 cm in diameter, the mass over 3,5 kg with excellent homogeneity. Residual self-absorption has been improved by factor 2-4 by annealing[3]. The state-of-art properties of such YAP:Ce single crystal scintillators include light yield of 25,000 ph/MeV, energy resolution ≤5.5% for 662 keV γ-rays from a ^{137}Cs source and absorption length >25 cm.

The enhanced technology of single crystal preparation has matured in production with high yield that enables cost-competitive alternative to traditional scintillation materials such as NaI:Tl with the advantage that YAP:Ce is not brittle and does not need encapsulation. It can easily replace ordinary dose-meters in harsh or chemically agressive environment or in new window-free detectors as YAP:Ce can be in direct contact with analyte (liquid or gas), e.g. in well detectors of radioactive liquids, large-window α or β detectors and spectrometers or X-ray applications.

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P.21

Spectroscopic, luminescent, spectral-kinetics and thermal-physical properties of CaF_2-SrF_2:Tm and CaF_2-SrF_2:Ho single crystals for IR lasers

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POSTER

Two-micron solid state lasers are of great interest for some medical surgery, lidar systems, gas (NH_3, CO_2, CO) detection, as a pump source for nonlinear materials to generate mid-IR radiation et al. Fluoride materials are also very promising materials for laser devices, because they have high transmission in a wide spectral range (from 0.16 to 11 μm), low phonon energy, high thermal conductivity, clustering effect et al.

In the present paper, we investigated spectral-luminescence and spectral-kinetics properties of CaF_2-SrF_2:Tm and CaF_2-SrF_2:Ho single crystals. Fluoride crystals were grown by vertical directional solidification (Bridgman technique). We measured the thermal conductivity (50-300 K), refractive index, thermal coefficient for a series of single crystals.

For CaF_2-SrF_2:Tm and CaF_2-SrF_2:Ho crystals the absorption cross sections for $^3H_6 \rightarrow {}^3H_4$ and $^5I_8 \rightarrow {}^5I_7$ transitions, luminescence and gain cross sections for $^3F_4 \rightarrow {}^3H_6$ and $^5I_7 \rightarrow {}^5I_8$ transitions were estimated. Cross-relaxation processes in CaF_2-SrF_2:Tm solid solutions were studied. The mechanisms of upconversion luminescence of CaF_2-SrF_2:Ho crystals from $^5F_3$, $^5S_2(^5F_4)$, $^5F_5$ and $^5I_6$ level upon excitation of $^5I_7$ level of Ho^{3+}ions were investigated.

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P.22

Down-conversion IR luminescence in fluoride matrices doped with Pr^{3+}-Yb^{3+}, Eu^{3+}-Yb^{3+} and Ce^{3+}-Yb^{3+} ions pairs in CaF_2 and SrF_2 submicron disperse powders

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POSTER

One of the emerging applications of nanosized dispersed luminescent materials is the enhancement of solar panels efficiency by down-conversion of solar light to the spectral range where emission is absorbed by silica with the highest intensity.[1] This approach uses energy transfer processes and here fluoride based matrices are appear to be advantageous as low phonon energy provide lower excitation losses. The fluorite type crystals CaF_2 and SrF_2 are prospective candidates as host materials, because they have affinity to silicon lattice. Here we discuss the results of comprehensive work on investigation of down-conversion through energy transfer to Yb^{3+} ions from various donors: Pr^{3+}, Eu^{3+} and Ce^{3+}.

The samples were prepared by well-known co-precipitation technique from aqueous solutions.[2] All samples show good crystallinity, homogeneous phase distribution, and distribution coefficient about unity. The effect of high temperature annealing of samples is discussed here as it plays significant role on structural and luminescent properties samples.

In this work we show that energy transfer from Pr^{3+}, Eu^{3+} and Ce^{3+} to Yb^{3+} ions goes through different mechanisms. We discuss peculiarities of down-conversion luminescence for the studied compounds originating from complex character of heterovalent cations substitution. The data on spectral-kinetic properties of the samples, as well as external quantum yield of down-conversion luminescence are presented. Overall we show that fluorite type crystalline compounds doped with Pr^{3+}-Yb^{3+}, Eu^{3+}-Yb^{3+} and Ce^{3+}-Yb^{3+} ions pairs synthesized by simple and useful co-precipitation technique are prospective for using for enhancement of silicon solar cells efficiency.

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Bright luminescent diamond- fluoride composites for photonics

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Recent experiments on free-electron lasers, which may generate coherent electromagnetic radiation in the X-ray range, showed the tendency of increasing power of such lasers[1]. Thus, there are new challenges in fabricating novel detectors and visualizers for the “hard” high-power radiation. Fabrication of the detectors and visualizers based on the materials of the novel type, such as luminescent diamond-fluoride composites, is an important task for practical applications in X-ray optics. In order to expand the spectral range of diamond luminescence materials and to increase the intensity of X-ray luminescence (XRL), successful attempts were made to introduce europium fluoride in polycrystalline diamond[2]. Despite the demonstrated success, the signal-to-noise ratio was low due to using of individual europium compounds, which leads to luminescence quenching. Using of the Eu-doped compounds, such as hexagonal $NaGdF_4$, rather than a pure compound result in the increase of luminescence intensity.

Photo- and X-ray luminescent composites based on NaGdF4:Eu nanoparticles embedded in polycrystalline diamond (PCD) were obtained by CVD overgrowth technique. Both photoluminescence (PL) and XRL spectra reveal an intensive signal from Eu ions near 612 nm. The full width at half maximum (FWHM) of the peak is as low as 2 nm and 6.1 nm for PL and XRL, respectively. Photoexcitation at 395 and 256 nm wavelengths demonstrated the efficient energy transfer from Gd matrix to Eu anions. The use of β-$NaGdF_4$:Eu nanopowders instead of pure europium fluoride allow obtaining a high-intensity luminescence with a rather higher signal-to-noise ratio. PL mapping showed high density of XRL sources, which makes possible using a diamond-fluoride composite as high-resolution luminescent screens.

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P.24

POSTER

LPE grown of the thermoluminescent detectors based on the $Lu_{3-x}Gd_xAl_5O_{12}$:Ce/YAG:Ce epitaxial structures

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This work is dedicated to the development of new types of composite thermoluminescent (TL) detectors for simultaneous registration of the different components of mixed ionization fluxes based on the single crystalline films (SCFs) of Ce^{3+} doped $Lu_{3-x}Gd_xAl_5O_{12}$:Ce (x=0-1.5) garnet and $Y_3Al_5O_{12}$:Ce (YAG:Ce) substrates using the Liquid Phase Epitaxy (LPE) growth method. The possibility of creation of such type composite TL detectors using the differences between the TL glow curves, recording from the SCF and substrate parts of the composite detector in the conditions of α- and β-particle excitation, has been shown by us in [1].

The SCFs of $Lu_{3-x}Gd_x Al_5O_{12}$:Ce garnet with Gd concentration from 0 up to-2.5 have been recently successfully crystallized by the LPE method onto undoped YAG substrates from the melt-solutions based on the $PbO-B_2O_3$ flux, and their optical properties have been investigated as well [1]. In the present work we want to use the LPE grown epitaxial structures containing $Lu_{3-x}Gd_xAl_5O_{12}$:Ce (x=0-1.5) SCFs with different Gd concentration and Ce^{3+} doped YAG:Ce substrate for simultaneous registration of α- and β-particles. For this purpose, the TL properties of the mentioned epitaxial structures were examined in Risø TL/OSL-DA-20 reader under excitation by α- and β- particles of ^{242}Am and ^{90}Sr-^{90}Y sources.

We expect that the cation engineering of SCF content can result in more significant separation of the TL glow curves of SCFs and substrates in comparison with the prototype of such composite detectors based on the LuAG:Ce/YAG:Ce epitaxial structure. The obtained results confirm our assumption that the difference between the TL glow curves of $Lu_{1.5}Gd_{1.5}Al_5O_{12}$:Ce SCFs and YAG:Ce substrates increases up to 120°C in comparison with a respective value of 80°C in the prototype based on LuAG:Ce/ YAG:Ce epitaxial structure.

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P.25

Plasmonic materials and metamaterials for VIS and NIR applications obtained by bottom-up methods

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Novel fields of photonics – metamaterials[1] and plasmonic materials[2] require new fabrication techniques. Two approaches towards materials with unusual electromagnetic properties will be presented: (ii) the NanoParticle Direct Doping (NPDD)[3,4,5] and the Directional Solidification of Eutectics (DSE).[6,7]

We will demonstrate how to utilize the melt-based crystal growth techniques for manufacturing such advanced photonic materials enabling doping dielectric matrices simultaneously with plasmonic nanoparticles and quantum dots or rare earth ions and self-organized eutectic composites with tunable Localized Surface Plasmon Resonances (LSPR) at visible and near infrared wavelengths.[7,8,9]

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POSTER

P.26

Self-assembled, highly-tunable narrowband optical filters and polarizers based on ZnO–$ZnWO_4$ eutectic composites

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POSTER

Recent novel material platforms, such as metamaterials,[1] are based on engineering structure and intrinsic material properties to achieve unique light and matter interactions. Such new approaches to artificial media led to the development of micro/nano-structured composites emulating unusual electromagnetic responses.[2] Recently, composites consisting of all-dielectric, transparent materials in which light does not interact with plasmons or phonons have been proposed as an alternative approach.[3] However, composite materials can be generally lossy due to scattering effects induced by the inhomogeneity at the interfaces between the different compounds. To overcome such problems, complicated and costly manufacturing procedures, like top-down approaches, are generally used. Here, we demonstrate ZnO–$ZnWO_4$ eutectic self-assembled all-dielectric composite, grown by the micro-pulling method, with a polarization and temperature dependent narrow-band transmission at 397 nm. Such an interesting optical response, not present in ZnO and $ZnWO_4$ single crystals, results from the orientation of the anisotropy axes of the two constituents, which self-organize in ordered structures.

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Single crystal growth and physical properties of MCo_2Al_9 (M = Sr, Ba, Eu)

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Single crystals of $SrCo_2Al_9$, $BaCo_2Al_9$ and $EuCo_2Al_9$ were grown using a self-flux method. The crystal structure was examined with x-ray powder diffraction measurements and the Rietveld refinement. Physical properties of the compounds were studied for the first time by means of electrical resistivity, magnetic susceptibility and heat capacity measurements. Measurements revealed that $SrCo_2Al_9$ and $BaCo_2Al_9$ both show diamagnetic behavior and $EuCo_2Al_9$ is an antiferromagnet with T_N = 3.5 K and effective magnetic moment of μ_{eff} = 7.86 μ_B. Magnetic susceptibility vs. magnetic field curve measured with magnetic field applied along the *c* direction has a complex step-like behavior. Heat capacity measurements confirm the transition temperature and its shift towards lower values with applied magnetic field, regardless of the measurement configuration. Resistivity measurements show metallic behavior of all of the three compounds, with no visible transitions.

P.28

POSTER

Growth mode, arrangement and polarity of GaN nanowires grown by PAMBE on Si(001) substrates: importance of SiN_x interlayer

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An unique feature commonly observed for GaN nanowire (NW) growth on silicon is that a thin (~2 nm) SiN_x film forms on the substrate as soon as the nitrogen source is switched on to start the NW growth. In this work we show that presence/absence of this SiN_x layer determines growth mode, polarity and arrangement of GaN NWs on Si(001). For that two samples S1 and S2 of GaN NWs were grown by PAMBE on Si(001) under the same conditions, except of substrate preparation. After initial nitridation additional substrate cleaning and a second nitridation step were applied for S1 while they were omitted for S2.[1]

SEM studies show that S1 contains uniform ensemble of NWs oriented perpendicularly to the substrate surface, i.e. similar to commonly observed on Si(111).[2] These NWs grow catalyst-free on a thin SiN_x layer as found from TEM analysis. TEM and XRD studies show that such NWs are N-polar and contain pure wurtzite phase with perfect *in-plane* as well as *out-of-plane* alignments to the Si substrate. On the contrary, two sets of NWs are found in S2: vertical as in S1 and novel ones, Ga-polar and perpendicular to the Si(111) planes, thus inclined to the substrate surface. They start growing as zb-GaN pyramids in a direct contact with the Si substrate. Then cubic-wurtzite transition takes place and pure wurtzite GaN inclined NWs grow from pyramids' facets.[3] This is confirmed by synchrotron-based Grazing Incidence X-Ray Diffraction (GIXRD) measurements showing, in addition to wurtzite GaN peaks, zb-GaN signal from S2.[1] Zb-GaN islands were too small to be observable by laboratory XRD, so grazing incidence geometry and intense synchrotron X-ray beam had to be used. Growth of inclined NWs in S2 is explained by presence of residual oxide that locally protect the substrate against SiN_x formation during the initial nitridation. Then, the oxide is dissolved in gallium and Ga-assisted growth of zb-GaN islands starts leading to Ga-polar inclined NWs. This is quite different from behavior of NWs in S1 when additional substrate cleaning steps remove residual oxide islands allowing creation of continuous SiN_x film on which N-polar vertical NWs uniformly grow. These findings show a crucial role the SiN_x film plays for arrangement and properties of GaN NWs on Si.

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P.29

Polarity of self-induced GaN nanowires on Si(111) studied by Kelvin Probe Microscopy: influence of Si substrate preparation

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Growth of GaN nanowires (NWs) having polar, wurtzite structure on nonpolar Si substrates raises the issue of the GaN NW polarity. Depending on the interface chemistry and the details of the growth procedure the coexistence of NWs with different polarities inside the NW ensemble has been observed.[1,2] Since the specific polarity affects optical and electronic properties of NWs, reliable methods for its control and characterization are needed.

In this work we use Kelvin probe force microscopy (KPFM) to asses polarity of GaN NWs grown by plasma-assisted MBE on Si(111) substrates. Complementary images of KPFM, namely topography and contact potential difference (CPD) were analyzed.[3] They allow measuring the polarity of individual NWs over an area of tens of μm^2 and provide statistics on the polarity of the ensemble with an accuracy hardly reachable by other methods. Our studies show that uniformity of polarity of GaN NWs on Si(111) strongly depends on the procedure used for substrate processing prior to the NW growth. As high as 20% of NWs with reversed polarity (i.e. Ga-polar) were found if the Si substrate was etched in diluted HF and then annealed in the growth chamber to remove hydrogen passivation prior to the substrate nitridation.[4] Despite the fact that such procedure leads to clean 7x7 substrate surface reconstruction some islands of residual oxide are apparently left that may induce growth of Ga-polar GaN NWs.[1] Additional substrate treatments (RCA etching, Ga cleaning, etc.) were tested. However, the best results, i.e. purely N-polar ensemble of NWs, were obtained on epi-ready Si wafers thermally deoxidized in the growth chamber at ~1000°C just prior to their nitridation. Interestingly, no mixed polarity was found for GaN NWs grown under similar conditions on Si(111) substrates covered by a thin amorphous Al_xO_y buffer layer.[5] This shows the crucial role the chemistry at the GaN/Si(111) interface plays for polarity of GaN NWs.

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POSTER

P.30

POSTER

Growth of bulk SiC crystals by the PVT method with nitrogen and cerium impurities

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Silicon carbide of 4H polytype (4H-SiC) is one of the most promising materials for power electronic and piezoelectronic applications in harsh environments thanks to its excellent electrical, chemical and mechanical properties. The nitrogen is a basic dopant in SiC which determines electrical and optical properties of SiC[1], while the cerium dopant is very rarely used dopant in SiC bulk crystals. There are only few papers[2-5] in which SiC crystals were grown by PVT method in the presence of Ce impurity.

In this work we present the results of structural and electrical properties investigations of bulk SiC doped with different nitrogen dopant content (0-10 wt%) added to the argon growth atmosphere, which were grown by PVT method in the presence, as well as without the cerium impurity. As the source of cerium, a commercial powder CeO_2 (0.5 wt%) was used. Growth surfaces and the crystalline structure were studied by: optical microscopy, atomic force microscopy, KOH etching, X-ray diffraction and secondary ion mass spectroscopy. The resistivity of crystals was evaluated by the contactless method at microwave frequencies at 300 K. The type of electrical conductivity and charge carrier concentrations were estimated from electrochemical C-V profiling.

In crystals obtained without the presence of cerium during the growth a mixture of 4H-, 6H- and 15R-SiC polytypes was observed, while for crystals grown with cerium impurity in sources, the 4H-SiC polytype uniformity was around 100%.

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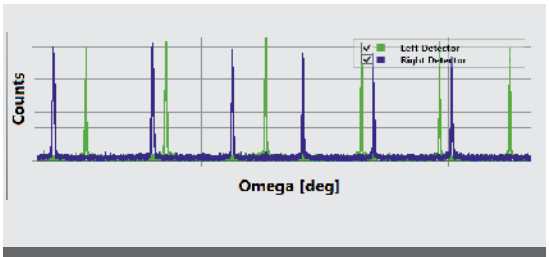

Omega-scan diagram (Sapphire)

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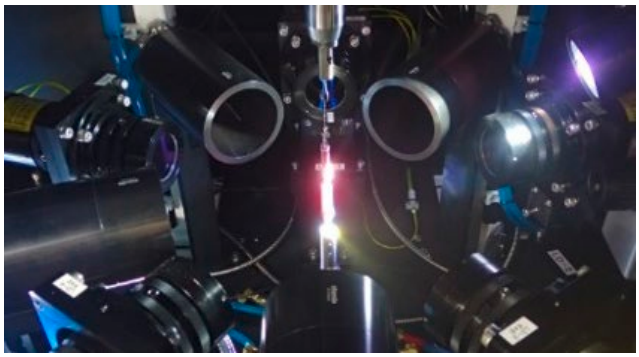

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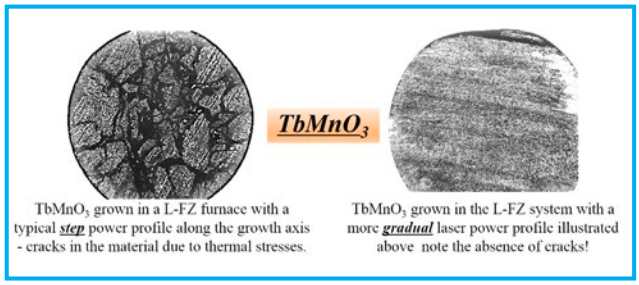

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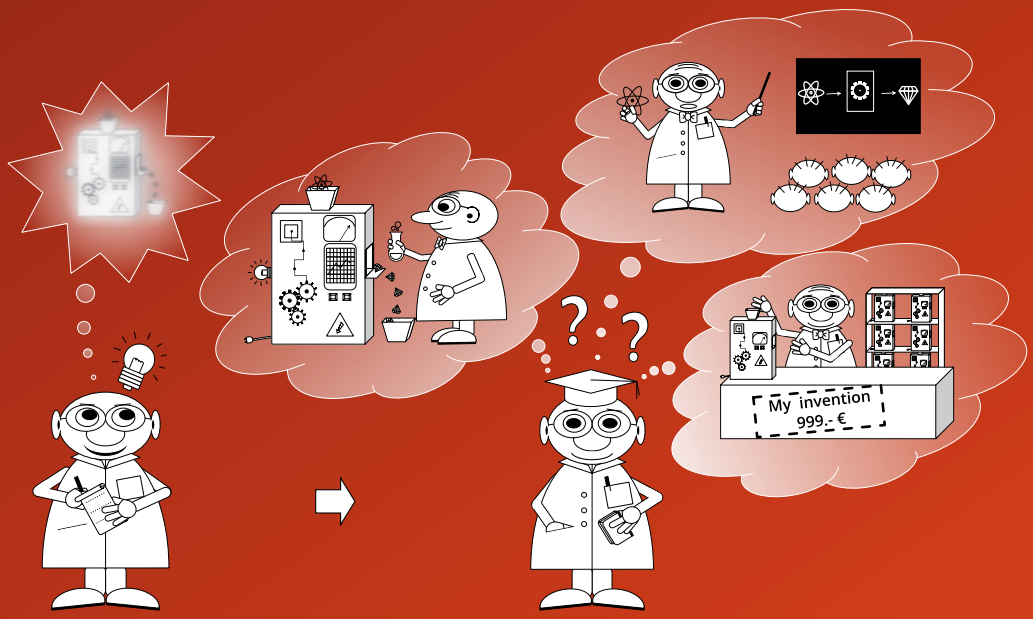

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