Crystal Growth Laboratory

Your Competent Partner in Crystal Growth and Solidification Processes

Annual Report 2007

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Crystal Growth Laboratory

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Crystal growth processes provide basic materials for many applications. The research and development of crystal growth processes is driven by the demands which come from the specific applications; but in general there is a need for an increase of crystal dimensions, improved uniformity of the relevant crystal properties in the micro and macroscale and materials with new properties.

Therefore, the focal area of research of the Crystal Growth Laboratory (CGL) is to develop – in close collaboration with industry - equipment and processes for the production of bulk crystals and thin films in order to meet the increasing requirements on crystal quality and cost reduction.

The strategy of CGL is to optimize the crystal growth processing by a combined use of experimental process analysis and computer modeling. These activities are based on a suitable experimental infrastructure and on highly efficient user friendly simulation programs. These computer codes, which are continuously further developed, are used for and by the industrial partners to develop crystal growth equipment and processes.

CGL was founded at the Department of Materials Science of the University of Erlangen - Nuremberg by Prof. Dr. Georg Mueller in 1979. Since 1996 the Crystal Growth Laboratory has established the working group "Crystal Growth" at the Fraunhofer Institute for Integrated Systems and Device Technology (IISB) in Erlangen. This working group became the Department Crystal Growth in autumn 1999. Since 2005 the CGL is also present in Freiberg/Saxonia within the Fraunhofer Technology Center for Semiconductors which is a common department of Fraunhofer IISB from Erlangen and Fraunhofer ISE from Freiburg.

Since the foundation of CGL more than 280 papers in scientific journals and conference proceedings have been published. Furthermore, CGL has educated a lot of experts in this field. Around 200 "Study" and diploma theses and around 40 PhD theses may serve as a reference for this.

More than 90% of funding of the Department of Crystal Growth result from research contracts directly with industrial partners or with public funding organizations like the Federal Ministry for Education and Research, the Bavarian Research Foundation. Since 1996, CGL at Fraunhofer IISB has acquired almost 15 Mio. Euro from the different sources indicated above.

Today, CGL consists of around 35-40 highly motivated coworkers. They are experts in different fields, like systems engineering, metrology, computer simulation, physics, material science, mathematics. In 2007 CGL has consolidated its position as world-wide acknowledged center of competence in the field of crystal growth.

In the field of crystallization of...
solar silicon CGL together with its subsidiary in Freiberg, the Fraunhofer Technology Centre Semiconductor Materials, has developed ways to avoid the formation of harmful precipitates and gained valuable knowledge about the heat and mass transport processes in the large silicon melts for its industrial partner by experimental analyses and numerical simulation. In the area of growth of optical crystals special Calcium Fluoride single crystals were grown which might serve as basis for future stepper generations. Furthermore the full automated growth of sapphire ribbons was demonstrated.

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In the field of manufacturing of gallium nitride from metallic solutions under ambient pressure conditions a process was developed which allows the production of several low defect GaN templates with up to 75mm diameter in one run. In the area of high power devices on the basis of silicon carbide the focus is put on the analysis of crystal defects which are harmful for the long time reliability of the devices. Thereby, measures which allow to avoid special crystal defects during the epitaxial process have been developed together with the industrial partners. This serves as basis for a better reliability of the devices.

In the field of modeling the software CrysMAS, which is the main product of CGL, was tailored further in order fulfill the needs of the customers with respect to easier usage and more efficient computation of fluid problems. The further developments allow among others to predict the influence of convection on the microstructure formation in metallic alloys during future solidification experiments onboard the International Space Station. In the frame of the Fraunhofer Profx² program a generic concept for coupling of different software tools was developed. This concept will be realized to couple CrysMAS to Openfoam, which has turned out to be very efficient for three dimensional fluid flow simulations.

Last but not least several invited talks during international conferences as well as the collaboration in different national and international expert panels in the field of crystal growth have contributed to strengthen the international reputation of CGL. Furthermore, CGL has organized several events. At this point it has to be mentioned especially the scientific colloquium on the occasion of the official retirement of the founder and former head of Erlangen’s Crystal Growth Laboratory Prof. Dr. Georg Müller. His outstanding contributions in the field of teaching were honoured in 2007 with the “good teaching award” of the Bavarian Ministry of Science, Research and Art.

Fig. 2: Prof. Dr. Georg Müller during the closing ceremony of the scientific colloquium on the occasion of his official retirement.
Photovoltaic is one of the economic branches, which has grown significantly in the last few years. This growth has a direct impact on the creation of new jobs. At the same time photovoltaic is one of the best answers to the greenhouse effect, which acts as one of the greatest threats to earth.

According to different market studies the rapid growth of photovoltaics in the last years will continue further despite of the shortage of the silicon raw material. Therefore, much more efficient methods for manufacturing of silicon wafers are required. Once this shortage of the silicon feedstock is handled efficiently, the demand for silicon wafers can quantitatively be met easily, for the production of solar cells. However, the material quality of the silicon wafers will decide the ranking of the manufacturers in comparison to their competitors.

The necessary silicon material for solar cells is manufactured by the principle of directional solidification with a market share of 75% in 2005. A silicon block is produced from the feedstock material by a melting and crystallisation process. After separating the block into columns the individual silicon wafers are produced by wire sawing.

The quality of the silicon wafers is extremely dependent on the heat- and mass transport occurring during the crystallisation and cooling process. Despite of the very well known fact that melt convection plays an important role for the heat and mass transport, the basic understanding about its mechanism and consequences to the properties of the silicon wafer is lacking.

The solidified silicon block is relative highly contaminated with carbon, oxygen, nitrogen and metallic impurities. Furthermore, these impurities lead to other crystal defects (Si$_3$N$_4$- and SiC-precipitates) and can interact among each other just like dislocation. These defects can reduce the charge carrier lifetime in the silicon wafer and lead to a lower efficiency of the solar cells. In addition, the precipitates can be responsible for short circuits in the solar cell and might trigger a total breakdown of the energy conversion.

The main sources for the incorporation of impurities into the silicon during the manufacturing of silicon wafers are the inclusion of carbon, oxygen, nitrogen and metallic impurities during the growth process. These impurities can lead to the formation of crystal defects, which can reduce the charge carrier lifetime and lead to a lower efficiency of the solar cells. In addition, the precipitates can be responsible for short circuits in the solar cell and might trigger a total breakdown of the energy conversion.
Correlation of the formation of precipitates in multicrystalline silicon with heat and mass transport processes

crystallisation process are graphite parts in the furnace and the Si₃N₄ coating of the SiO₂ crucible. The metallic impurities diffuse especially out of the crucible coating with the effect that the silicon block has a very low charge carrier lifetime in the border area which has to be castaway. In addition, the silicon nitride coating, which is sprayed on the fused silica crucible, is the main source for the nitrogen contamination.

The Technology Centre for Semiconductor Materials (THM) Freiberg started a close collaboration together with an industrial partner within the framework of the KoWäSto Project (P11096) (until 08/07) and the HiQaSil Project (12416/2025) (start 11/07) in order to improve the material quality for silicon wafers. THM is a combined branch of Fraunhofer IISB, Erlangen and Fraunhofer ISE, Freiburg.

Unlike normal industrial furnaces we have developed a special R&D furnace to vary individual process parameters very easily, to check the influence on the material quality. The experimental work has been accompanied by extensive model building and numerical simulation.

The observations show that the interior parts of the crystallisation furnace (insulating materials, crucible materials, crucible coating, and atmosphere) have a considerable influence on the creation of the precipitates.

By means of the numerical models it was possible to develop the so-called "virtual machine" which allows to describe the experimental results of the R&D furnace. The main tasks were the calculation of the stream-function and the mass transport in the melt.

Melt convection is assumed to be responsible for the C, N and O distribution whereas the O distribution is linked to the shape of the solid-liquid interface. Furthermore a quantitative description of the mass transport for C, O and N is also possible.

The current research activities are concentrated on the individual transport mechanism in the melt for the different precipitates. The aim of this work is to limit the occurrence of the precipitates and to reduce or even to eliminate them totally.

The research and development activities are funded partly by the European Regional Development Fund (ERDF) and by the ministry of economics and employment of the state of Saxony.

![substituierter Kohlenstoff](image)

Fig. 2: Carbon distribution measured with Fourier transformed IR-Spectroscopy (top)
Calculated stream-function for half of the crystallisation in the R&D-furnace (bottom)
Reduction of basal plane dislocations during epitaxy of 4H-SiC for high voltage devices

Silicon Carbide (SiC) is the preferred material for high power/high voltage applications because of its intrinsic physical properties. High voltage devices fabricated on SiC work more efficient than silicon based devices as power loss is reduced and they are more rugged.

In case of the unipolar devices already commercially available, crystal defects are regarded a limitation of the production yield. Concerning bipolar devices, the crystal defects are a risk for long-term stability. Therefore, such devices are not commercially available yet. The KoSiC project funded by the "Bayerische Forschungsstiftung" strives to deepen the comprehension of crystal defects to increase the production yield of unipolar devices and to render commercial use of bipolar devices possible.

The substrates contain different types of defects, which can propagate into the epitaxial layer. Further crystal defects can be generated during epitaxy or subsequent device processing – the problem covers the whole production process. The basic physical understanding for the properties and effect of defects on the device performance should be established with this project. From this knowledge the substrate and process requirements can be drawn for long-term stable bipolar devices. In case of unipolar devices the knowledge gained about defects will be used for increasing production yield and therefore a better competitive position and market penetration. To achieve the aims of the project, several skilled partners from industry and scientific research are needed with competences in all fields along the production path of SiC devices. The project partners besides the Fraunhofer IISB are SiCED Electronics Development, SiCrystal and Infineon Technologies as well as the Chair of Applied Physics of the University of Erlangen-Nuremberg.

Basal Plane Dislocations (BPD) are an example of the complex interaction between crystal defects and device reliability. BPDs are thought to influence the long-term stability of bipolar SiC based devices. This defect type occurs during crystal growth and the wafers cut from these crystals contain BPD densities in the range of \((10^3 - 10^5)\) per cm\(^2\). It can propagate.

![Fig.1: Typical etch pattern of a 4° off-axis 4H-SiC substrate. Different etch pit morphologies represent different defect types.](image)
Reduction of basal plane dislocations during epitaxy of 4H-SiC for high voltage devices

into the epilayer and therefore in the active area of bipolar devices. During device operation the BPD converts to a Stacking Fault, which is a very critical defect as it grows continuously. This leads to increasing forward voltage drop and at least to device failure.

One part of the KoSiC project is the reduction or avoidance of BPD in epilayers which can be achieved by a well-suited epitaxial growth process. The BPD density can be reduced by a dislocation conversion process depending on epitaxial growth conditions. The converted dislocations are regarded as of minor importance for device performance and stability. To establish such an epitaxial growth process, several epitaxial growth runs have been performed on 4° off-axis 4H-SiC substrates. The epilayers were characterized using defect selective etching, which is a very common method to investigate the dislocation content of crystalline samples. With this method, the intersection points of dislocations at the sample surface are decorated with characteristic etch pits, compare figure 1.

Varying single and multiple epitaxial growth parameters shows the influence of each parameter on the dislocation conversion. The results of these experiments are shown in figure 2. All of the BPDs from the substrate were converted during epitaxy for a certain combination of epitaxial growth parameters. The next step is the investigation of the long-term stability of bipolar devices based on those BPD-free epitaxial layers.

Fig. 2: Ratio of the BPD density of the epilayer and its corresponding substrate.

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Reduction of the dislocation density in LPSG-GaN

GaN is an important wide bandgap semiconductor for optoelectronic and electronic devices. A low dislocation density is an important factor for a high efficiency and a high lifetime of the devices.

The low pressure solution growth (LPSG) technique for the growth of GaN has been developed at Fraunhofer IISB. An important aspect for the layer growth is the achievement of a reduced dislocation density in comparison with the used template, a MOCVD-GaN layer on sapphire. Aim of the present investigation is the understanding of the mechanism for the reduction of the dislocation density. This knowledge can be used in order to optimize the growth conditions for GaN.

The investigation method in choice is here the Transmission Electron Microscopy (TEM). This method allows analyzing single dislocations. It is possible to discriminate the different types of dislocations by special imaging conditions.

From our results we can see, that layer growth starts with islands. The nucleation of the islands takes place on the template in a non ordered way. The islands have faceted surfaces, where these surfaces can be indexed by (1-10n).

The dislocations bend towards the facets of the islands. The bending angle, measured towards c-direction, depends on the angle of the facet and on the type (Burgers vector) of the dislocation. Figure 1 shows a TEM-image, where the dislocations are the darker lines. Different dislocations bending towards the same facet are visible. The dislocations with a-type Burgers vector bend by an higher angle than the dislocation with a+c-type Burgers vector at the same facet. Dislocations that bent off the main growth direction are more probable to annihilate. The dislocation density decreases within the first micrometer of LPSG-GaN-layer by one order of magnitude.

In order to explain the bending of the dislocations we calculated the line energy for the different types of dislocations in the possible glide systems. Figure 2 to shows an example for these calculations. Here the line energy is plotted for an a-type dislocation in the pyramidal glide system for three different facet angles. Comparing these three curves the minimum of

![Fig.1: TEM-Image showing the dislocation bending at the MOCVD-LPSG-GaN interface (white arrow) for dislocations (dark lines) with different Burgers vectors at the same facet.](image)
Reduction of the dislocation density in LPSG-GaN

the line energy shifts to higher angles for steeper facets. This means that steeper facets favour the banding to the basal plane. The angles where the line energy is minimum are the same as the bending angles determined experimentally.

These energetic considerations can explain the behaviour of the dislocations during LPSG GaN layer growth. By controlling the islands growth it is possible to bend the dislocations of the direction of the layer growth and to force the annihilation of the dislocations.

Acknowledgement:
The work was financially supported by the BMBF and the “Fraunhofer Doktorandinnen Programm”.

Fig. 2: Numerical calculation of the line-energy for a dislocation with a-type Burgers vector in the pyramidal glide system for different facets.
Solidification processes of metallic alloys have a very high importance in industry. Almost every component made of metal had at least one process of solidification during its life cycle. Depending on its further processing all or at least some properties of the component are defined by the casting process. The physical phenomena found in the mushy zone during the solidification process are of great complexity. Technical alloys solidify usually with dendritic interface morphology as shown in figure 1.

In this interface area between the melt and the solid a considerable number of phases and structures are formed, which define the later properties of the material. For example the spacing between the primary ($\lambda_1$) and the secondary ($\lambda_2$) dendritic arms defines the distance between precipitates, which may form out of the alloy components in the melt and constitute between the arms. This distance of the particles determines the mechanical strength of many alloys. Figure 2 shows these processes in a sketch.

The numerical modeling of these processes would make it much easier to guaranty specific material properties of a component. To get closer to this objective Fraunhofer IISB and European partners work together within the framework of the MICAST project on the analysis and modeling of the microstructure formation. In these studies the so-called directional solidification method is used. This has the advantage that the formed microstructure is also directed and therefore ordered. This facilitates or even enables a quantitative analysis of important parameters such as the primary dendritic arm spacing. Figure 3 shows the process conditions for a typical specimen.

An important topic within the MICATS project is the influence of convection to this forming microstructure. For example it is investigated the change of the primary and secondary dendrite spacing if the melt is artificially stirred by a rotating magnetic field during directionally solidification. This type of time dependent magnetic field acts like an invisible spoon to the melt causing a rotating primary flow with secondary flows at the solid-liquid interface.

While other partners investigate this by ground based or microgravity experiments, in our group we try to integrate models for describing alloy solidification in our software CrysMAS. Recently a multi-scale model was implemented to calculate directional alloy...
Multi-scale modeling of the microstructure formation during directional solidification of alloys

solidification under the influence of forced melt flow. The models allow detailed calculations of the microstructures in the specimen, which is mainly characterized by the primary, the secondary dendritic arm spacing and changes in the locale concentration of the alloying elements caused by melt flow. Nevertheless the software can capture the thermal characteristics of a complete laboratory furnace in a so called global model.

Figure 4 shows a typical result of such fully time dependent calculations. Shown is the area of the mushy zone during the solidification process of an aluminum alloy with 7wt% of silicon. The arrows visualize the secondary flow field cause by the RMF. Due to this stirring, alloying elements, in this case silicon, are piled up at the centre of the sample (left side color code) like unsolved sugar in a stirred cup of tea. Due to this change in concentration, which locally changes the melting temperature of the alloy, a liquid channel is formed at the symmetry line of the sample. The color code on the right side of figure 4 gives the fraction of the melt in the mushy zone. This formation of a channel under the influence of a RMF was also found in experiments conducted by MICAST partners.

At present times the calculated microstructure parameters (primary and secondary dendrite spacing) are correlated to experimental values and it is investigated if the models have to be modified to capture the physics of the solidification process.

If this work is done the software should serve as tool for scientist to test experimental condition or furnace designs if they are advantageous for objectives of their experimental studies. This shall help to reduce the risk of failure of e.g. expensive space experiments.

Acknowledgement: The introduced assignment is promoted by project 50WM0546 of the DLR and by project 1437-00-NL-SH of the ESA within MICAST III.
Publications

2007

G. Müller
The Czochralski Method - where we are 90 years after Jan Czochralski’s invention

J. Friedrich
Control of melt convection in VGF and CZ crystal growth configurations by using magnetic fields: Theory and examples

G. Müller
Fundamentals of Melt Growth

D. Vizman, J. Friedrich and G. Mueller
3D time-dependent numerical study of the influence of the melt flow on the interface shape in a silicon ingot casting process

D. Vizman, M. Watanabe, J. Friedrich and G. Müller
Influence of different types of magnetic fields on the interface shape in a 200 mm Si-EMCZ configuration
Journal of Crystal Growth 303 (2007) 221-225

G. Sun, E. Meissner, P. Berwian, G. Müller, J. Friedrich
Study on the kinetics of the formation reaction of GaN from Ga-solutions under NH₃ atmosphere

J. Fainberg, D. Vizman, J. Friedrich and G. Mueller
A new hybrid method for the global modeling of convection in CZ crystal growth configurations

C. Reimann, J. Friedrich, G. Müller, S. Würzner, H.J. Möller
Analysis of the Formation of SiC and Si3N4 Precipitates During Directional Solidification of Multicrystalline Silicon for Solar Cells

G. Müller, J. Friedrich (Editors)

J. Dagner, J. Friedrich, G. Müller
Simulation of the microstructure formation in Al-Si alloys by multi-scale modeling of directional solidification
Proc. of Solidification Processing 2007 (SP07) (2007)121-125

B. Kallinger, E. Meissner, D. Seng, G. Sun, S. Hussy, J. Friedrich, G. Müller
Study on the sublimation growth of GaN using different powder sources and investigation on the sublimation behaviour of GaN powder by means of thermogravimetry
phys. stat. sol. (c) 4, No. 7 (2007) 2264-2267

G. Sun, E. Meissner, S. Hussy, B. Birkmann, J. Friedrich and G. Müller
Morphologies of GaN single crystals grown from Ga solutions under flowing ammonia
Publications

2006

B. Birkmann, S. Hussy, G. Sun, P. Berwian, E. Meissner, J. Friedrich, G. Müller
Considerations on facetting and on the atomic structure of the phase boundary in low-pressure solution growth of GaN

G. Müller, J. Friedrich
Yield Improvement and advanced defect control - driving forces for modeling of bulk crystal growth

B. Birkmann, C. Salcianu, E. Meissner, S. Hussy, J. Friedrich, G. Müller
Characterisation of the electrical properties of solution-grown GaN crystals by reflectivity and Hall measurements
phys. stat. sol. (c) 3 No.3 (2006) 575-578

S. Steinbach, J. Dagner, M. Hainke, J. Friedrich, L. Ratke
A Combined Numerical and Experimental Study of the Effects of Controlled Fluid Flow on Alloy Solidification

S. Hussy, E. Meissner, B. Birkmann, I. Brauer, F. Scholz, H. P. Strunk, G. Müller
Morphology and microstructure of a-plane GaN layers grown by MOVPE and by low pressure solution growth (LPSG)

Sulfur incorporation into copper indium diselenide single crystals through annealing in hydrogen sulphide
JOURNAL OF APPLIED PHYSICS 99 (2006) 043502

J. Dagner, M.Hainke, T.Jung, M. Kellner, H. Hadler, J. Friedrich, G. Müller
Global Modeling of Heat Transfer during Solidification Experiments

M. Hainke, J. Dagner, M.Wu, A. Ludwig
Control of Interdendritic Convection by the Application of Time-Dependent Magnetic Fields during Directional Solidification of Aluminum Alloys,

M. Hainke, J. Dagner, J. Friedrich, G. Müller
Simulation of ESA’s MSL Furnace Inserts and Sample-cartridge Assemblies using the Thermal Model Tool CrysVUn

M. Hainke, S. Steinbach, J. Dagner, L. Ratke, G. Müller
Solidification of AlSi Alloys in the ARTEMIS and ARTEX Facilities Including Rotating Magnetic Fields – A Combined Experimental and Numerical Analysis

J. Dagner, A. Weiss, M. Hainke, G. Zimmermann, G. Müller
Global Modeling of Directional Solidification of Aluminum Alloys using the Software Package CrysVUn
Equipment

Laboratory space
200 m² laboratory space in total at university and Fraunhofer IISB plus offices

Crystal growth
- several multi-zone furnaces for vacuum and high pressure conditions (for 2" - 6" crystal diameter)
- OKZ/300-100 for the growth high melting oxides
- several multi zone furnaces for sample preparation and growth of small diameter crystals

Analysis and characterization of materials
- Several optical/infrared microscopes
- Access to high resolution microscopes
- Mapping system for optical spectroscopy of semiconductor wafers
- Interferometric profilometer for surface analysis of semiconductor wafers
- X-ray Laue camera
- Hall-measurement-system (temperature dependent 15K-650K)
- Measurement system for characterization of deep and shallow levels by capacitance techniques (CV, DLTS) and by conductance techniques (TSC, PICTS)
- Differential Thermal Analysis for determination of phase diagrams
- Differential Scanning Calorimeter for thermodynamic and kinetic studies
- Thermogravimetry

Preparation and metallography
- Facilities for preparative work related to wafer preparation (grinder, annular and wire saws, lapping and polishing equipment)
- Several evaporation systems
- Sputtering systems (DC, 6" target diameter)

NETZSCH-STA 449 Jupiter and gas installation at CGL
Contact and travel information

Contact

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Travel Information

By car
Use Autobahn A3, exit Tennenlohe, follow signs for Erlangen, after 2 km take exit for "Universität Südgelände", then follow signs for IISB: 1.6 km north on Kurt-Schumacher-Straße, then turn left twice into Cauerstraße and Schottkystraße.

By plane
From Nürnberg (Nuremberg) airport use taxi (15 minutes) or bus 32 to Nürnberg-Thon and then bus 30/30E to Erlangen-Süd (30 minutes).

By train
From Erlangen station, use taxi (15 minutes) or bus 287 to Stettiner Straße (30 minutes). Convenient train services from Nürnberg Hauptbahnhof (central station) to Erlangen station.

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