

Marshall Plan Scholarship Paper

Electron Channeling Contrast Imaging Investigations of III/V Semiconductor Materials and Devices

Dipl. Ing. Stefan Kirnstötter BSc.



Sep 1st 2014 – March 1st 2015

Supervisor at Yale University

Associate Professor Minjoo Larry Lee PhD

Supervisor at Graz University of Technology:

Professor Peter Hadley PhD

15 Prospect Street, New Haven, Connecticut, United States of
America

Contents

| | |
|--|----|
| Abstract | 3 |
| Kurzfassung | 4 |
| Acknowledgements | 6 |
| Introduction..... | 7 |
| Chapter I: Fundamentals, Background and Analytical Techniques | 9 |
| Electron Channeling Contrast Imaging | 10 |
| Historical development of the analytical measurement technique ECCI..... | 11 |
| Setup and basic working principle..... | 12 |
| Channeling signal generation | 14 |
| Applications – Motivation | 16 |
| Simulation of Electron Channeling Patterns | 18 |
| Electron Beam Induced Current | 20 |
| Transmission Electron Microscopy..... | 22 |
| Etch pit density..... | 24 |
| Chapter II: Experimental Results and Interpretation | 25 |
| Gallium nitride (GaN) – as grown layers and light emitting diodes (LEDs) | 27 |
| Quantum dot (QD) and quantum well (QW) laser structures..... | 29 |
| Misfit dislocation investigations – GaAs on GaP/Si..... | 33 |
| InAlAs graded buffer..... | 36 |
| Direct 1-to-1 EBIC-ECCI comparison..... | 39 |
| Direct 1-to-1 EPD - ECCI comparison..... | 41 |
| Anti-phase domains..... | 43 |
| ECCI-measurement accuracy over several orders of magnitude | 45 |
| GaAs-Solar Cells on GaP and GaP/Si..... | 47 |
| Chapter III: Summary, Discussion and Outlook | 49 |
| Summary and Discussion..... | 50 |
| Future Studies | 51 |
| Bibliography..... | 53 |

Abstract

This extended paper reports about the scientific work performed by Stefan Kirnstötter during his six months research stay at Yale University in the group of Associate Prof. Minjoo Larry Lee. Most of this work was performed together as a team with Kevin Nay Yaung, a senior graduate student in the group of Prof. Minjoo Larry Lee. The initial goal of this research stay was the verification/initialization of the measurement method Electron Channeling Contrast Imaging (ECCI) at Yale University to investigate as grown III/V semiconductor materials as well as processed semiconductor devices. The focus was, to get a better understanding of the measurement principles. The forward and backscatter geometry have been compared by using forward scatter diodes and a pole piece mounted backscatter electron detector. To get a better understanding about the impact of the channeling conditions the samples were oriented in various (220), (400) or (260) orientations. Like mentioned in literature the highest symmetry channeling conditions resulted in the best backscatter electron contrast to visualize defects like dislocations. Therefore the backscatter geometry was superior to the forward scatter setup. Mounting the specimen in a 90° angle to the incoming beam results in an adjustment to the (001) center/channeling condition. This is way more difficult in the high angle symmetry, where the specimen is tilted ~70° as a starting conditions to increase the channeling yield. Besides the optimization of the ECCI setup and measurement routine itself, the measurement results have been compared with other analytical techniques like transmission electron microscopy (TEM), etch pit density (EPD) or electron beam induced current (EBIC). Threading or misfit dislocation densities (TDD, MDD), obtained with the different methods have been measured and statistically analyzed. It will be shown that multiple measurements on different areas are necessary to get statistically significant values for the dislocation density (especially in inhomogeneous distributed samples). Direct 1-to-1 comparisons of the same area on a GaAsP sample have been performed with EBIC, ECP and ECCI measurements. This can show that the dislocations are present on the same positions even though when they are sharp points in ECCI, dark round areas in EBIC and etched inverted pyramids in EPD. The reliability and the pros and cons of these analytical techniques are compared with each other.

Kurzfassung

Dieser Bericht spiegelt die wissenschaftliche Arbeit von Stefan Kirnstötter während der 6 Monate seines Forschungsaufenthalts an der Yale University in der Gruppe von Assoc. Prof. Minjoo Larry Lee wieder. Der Großteil der Arbeit wurde zusammen mit Kevin Nay Yaung, Doktorand in der Arbeitsgruppe von Prof. Lee durchgeführt. Das ursprüngliche Ziel dieses Forschungsaufenthalts war, die Überprüfung/Initialisierung der Messmethode Electron Channeling Contrast Imaging an der Yale University, um per Molekülstrahlepitaxie gewachsene III/V - Halbleitermaterialien sowie prozessierte Bauteile zu untersuchen. Am Anfang war der Fokus darauf gerichtet ein besseres Verständnis des Messprinzips und der Physik hinter dieser analytischen Methode zu bekommen. Zu allererst wurden beide in der Literatur verwendeten Messgeometrien verglichen: die Vorwärts- und Rückstreugeometrie. Hierfür wurden einerseits Vorwärtsstredioden und andererseits ein am Polstück des Rasterelektronenmikroskops befestigter Rückstreuelektronendetektoren verglichen. Um ein besseres Verständnis über die Auswirkungen der Channeling-Bedingungen zu bekommen wurden die Proben in verschiedenen Orientierungen ausgerichtet: z.B. in unterschiedlichen Variationen der (220), (400) oder (260) Orientierung. Wie in der Literatur erwähnt, erreichte die höchste Symmetrie Bedingung auch den besten Rückstreuelektronenkontrast um Versetzungen zu visualisieren. Hierbei ist die Rückstreu- der Vorwärtsstreugeometrie überlegen. Durch eine Montage der Probe in einem Winkel von 90 ° zum einfallenden Strahl, richtet man die Probe an das (001) Zentrum aus. Es ist um ein Vielfaches schwieriger die Probe in der Vorwärtsstreugeometrie so auszurichten. Neben der Optimierung des ECCI Aufbaus und der Messroutine selbst, wurden die Messergebnisse mit anderen analytischen Techniken, wie Transmissionselektronenmikroskopie (TEM), Ätzgruben-Dichte (EPD) oder Elektronenstrahl-induzierter Strom (EBIC) verglichen. Es wurden die Versetzungsdichten (TDD, MDD) mit den verschiedenen Verfahren bestimmt, verglichen und statistisch ausgewertet. Es wird gezeigt, dass mehrere Messungen an verschiedenen Bereichen erforderlich sind, um statistisch sichere Werte für die Versetzungsdichte (insbesondere in Proben mit inhomogener Fehlstellenverteilung) zu erhalten. Direkte 1: 1 Vergleiche von einem Gebiet auf einer GaAsP Probe wurden mit EBIC, ECP und ECCI-Messungen durchgeführt. Dies zeigt deutlich, dass die Versetzungen an den exakt gleichen Positionen sitzen und mit jeder Untersuchungsmethode

dargestellt werden können. Obwohl die Versetzungen in ECCI durch kleine Punkte, in EBIC durch dunkle runde Bereiche und bei EPD durch invertierte Pyramiden visualisiert werden. Die Methoden werden miteinander verglichen und es werden die Vor und Nachteile der jeweiligen Techniken dargestellt und verglichen. Ebenso wird auf mögliche Gründe für Diskrepanzen bei den 1-zu-1 Vergleichen eingegangen.

Acknowledgements

First of all I want to thank Assoc. Prof. Minjoo Larry Lee for his great scientific support and the kind way of including me in his research group.

I also want to thank Prof. Peter Hadley PhD from Graz University of Technology for his helpful support.

Most of the work shown in this report, was performed as a team together with Kevin Nay Yaung. Thanks for your patience and the fun time during our measurement stays.

I also have to thank the whole Lee-group at Yale Josef, Daehjuan, Michelle, Chris, Jukun, Taizo, for their help and warm way of welcoming me in their group for the time of my research stay.

Thanks to Martin for supporting Kevin and me with the measurements in the last month of my stay.

Special thanks go to Vivian Smart for helping me with a lot of office paper work.

Introduction

This report will give a review about the six months research work of Stefan Kirnstötter at Yale University at the Institute of Electrical Engineering under the supervision of Assoc. Prof. Minjoo Larry Lee.

The main goal of this work was to set up an electron channeling contrast imaging (ECCI) setup at Yale University and evaluate ECCI as an analytical technique to determine threading dislocation densities or investigate defects in III/V semiconductor materials and solar cells or other device structures in general.

There are various reasons why ECCI is an interesting alternative to already well established techniques to investigate semiconductor materials and devices. It is a very rapid method that needs no specimen preparation. It is very powerful and accurate. It is performed in a scanning electron microscope and besides a stage to adjust the sample it additionally only needs a backscatter electron detector or forward scatter diodes to visualize a channeling contrast. The measurement is rather cheap. It is non-destructive so after an ECCI investigation still other analytical techniques can be performed with the sample. No built in electric field, like a *pn*-junction or a Schottky contact are necessary.

Already well established methods like electron beam induced current, transmission electron microscopy or etch pit density investigations to determine the dislocation density are compared with ECCI results. Pros and cons of all these methods are taken into account and are compared with each other. Direct 1-to-1 comparisons of ECCI with other analytical techniques are presented. To the best of the authors knowledge this is the first time these methods are compared 1-to-1 with ECCI.

Various III/V semiconductor materials (GaN, GaP, GaAs, GaAsP, ...) and structures (buffers, transistors, light emitting diodes, lasers) grown by molecular beam epitaxy have been investigated and are presented in this report. III/V solar cells are of great interest in the scientific community because of their high efficiencies and good reliability. Historically ECCI was mainly used to investigate grain boundaries in metals, also several investigation on GaN have been published the last decade. Only very recently few groups used this powerful method to investigate defects in semiconducting samples.

In this work ECCI studies of different III/V materials and devices are shown. In the studies the growth conditions or substrates have been changed and the defect types and densities have been investigated. ECCI measurements of anti-phase domains are presented. The high resolution and good applicability of ECCI for samples with agglomerations of dislocations is pointed out.

Chapter I: Fundamentals, Background and Analytical Techniques

Electron Channeling Contrast Imaging

Electron Channeling Contrast Imaging is an analytical technique used to investigate defects, like dislocations (misfit or threading dislocations) or grain boundaries, in semiconducting materials and metals. ECCI investigations are performed in a scanning electron microscope using high acceleration voltages and beam currents. The determination of the density and type of dislocations in semiconductor materials can be of great importance. In comparison to transmission electron microscopy which is the most common method to investigate dislocation densities, no specimen preparation is necessary. ECCI enables the investigation of much bigger areas than it is possible with TEM. Additionally ECCI specimens are closer to the bulk like samples than the very thin electron transparent TEM specimens. Electron channeling patterns (ECP) are recorded to orient (tilt and rotate) the specimen in the required channeling conditions. The imaging can occur in specific diffraction conditions fulfilling standard invisibility criteria used during TEM investigations. ECCI investigations can be performed in high and low tilt geometry with a variety of backscatter detectors. In this work only single crystalline semiconductor materials and devices have been investigated. This chapter should explain the fundamentals and provide important background information about ECCI. The historical development of the analytical measurement technique during the last decades is reviewed. The necessary setup and basic working principle are explained in detail. The signal generation concept of channeling electrons is explained and potential applications and simulations are presented.

Historical development of the analytical measurement technique ECCI

In 1967 Coates et al saw Kikuchi lines while investigating single-crystals in a SEM (Coates, 1967). He saw an angular dependence of the patterns when zooming to low magnifications. The mechanism to produce the patterns was referred as electron channeling and he mentioned that it could be used to identify the orientation of the crystal. Nowadays Electron Back Scatter Diffraction (EBSD) is the preferred method for orientation and phase mapping (very often used in geology) in comparison to ECP because there is no need for specimen or beam rocking (for selected area channeling (SACP)). Also in 1967 the group of Brooker et al. predicted that it should be possible to monitor sub-grain boundaries and single dislocations in high resolution using electron channeling (Booker, G. R. et al., 1967). Shortly afterwards Clarke (Clarke, 1971) and Stern (Stern, R. M. et al., 1972) published the first experimental images of dislocations using electron channeling. In 1999 Simkin and Crimp (Simkin & Crimp, 1999) showed that dislocation imaging was also possible in low tilt (backscatter) geometry using modern field emission gun electron microscopes and high collection efficiency backscatter electron detectors. Before that, the high tilt (forward scatter) geometry was used to perform electron channeling experiments. In the 1990 several ECCI studies have been published showing near surface dislocations in Ga, Ni and Si (Czernuszka J. T. et al., 1990) using the invisibility criteria for screw dislocations. Also misfit dislocations in $\text{Si}_{1-x}\text{Ge}_x$ have been investigated by Wilkinson et al (Wilkinson, A. J. et al., 1993). A big part of the ECCI research community concentrates on studying metals imaging deformations twins, grain boundaries or twin grain boundaries (Gutierrez-Urrutia, I. S. et al., 2010), (Bieler, T. R. et al. , 2009) and (Simkin, B. A. et al., 2003). Recent studies concentrated on semiconducting materials like GaN or SiC. Picard et al ((Picard, Y. N. et al., 2007) and (Picard, Y. N. et al., 2007)) and Trager-Cowan et al (Trager-Cowan, C. et al., 2007) use a combination of EBSD and ECCI and published several studies visualizing dislocations and atomic steps.

Setup and basic working principle

ECCI investigations can be performed in low and high tilt geometry. In Figure 1 the high tilt ECCI measurement geometry is shown. This is the measurement geometry that was used exclusively before 1999 when Simkin et al (Simkin & Crimp, 1999) showed that low tilt investigations are possible too. Here the sample is tilt in an angle of $60^\circ - 80^\circ$. Forward scatter diode detectors are used to detect the channeling electrons containing the electron channeling information.

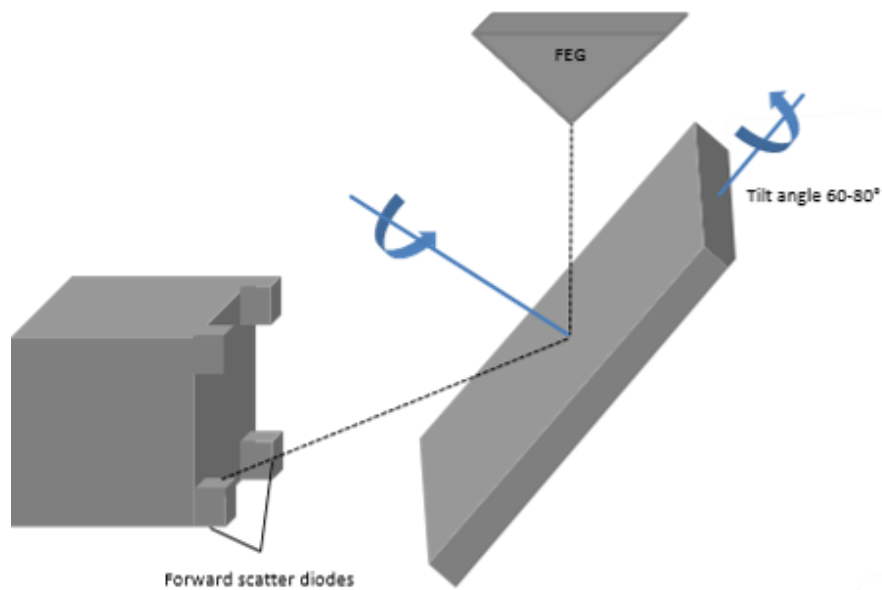


Figure 1: High tilt Electron Channeling Contrast Imaging measurement geometry using forward scatter diodes to detect the channeling contrast signal.

Figure 2 shows the low tilt ECCI measurement geometry. Here the sample is positioned very close underneath the backscatter detector that is mounted underneath/around the electron gun pole piece. This was the preferred measurement geometry during the investigation performed in this work.

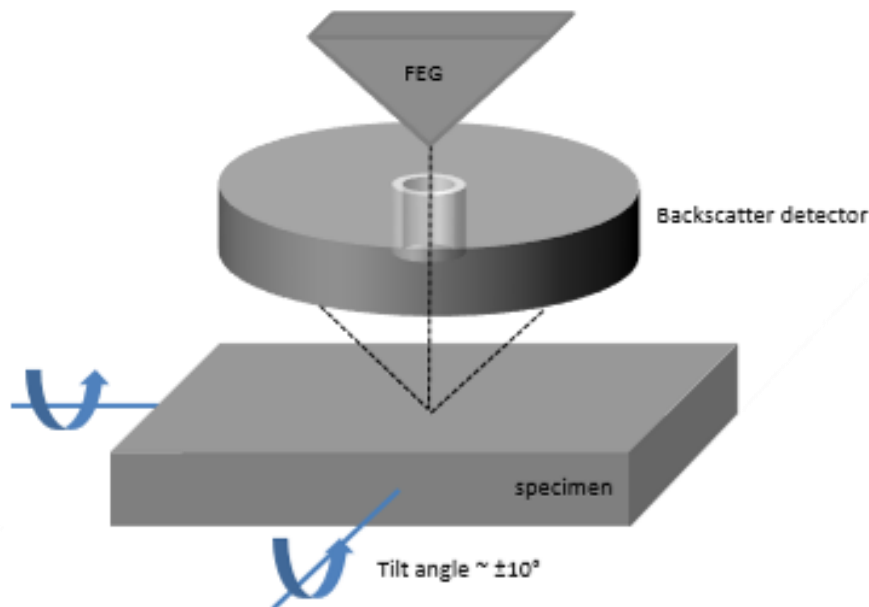


Figure 2: Low tilt ECCI measurement geometry using a pole piece mounted detector very close to the specimen surface to detect the BSE signal.

Electron microscopes equipped with a field emission electron gun are necessary to perform ECCI measurements. Other important measurement parameters are the electron beam current, it should be above 2 nA. In this work the best results have been archived when even higher values in the range of 5-30 nA have been used. The electron beam acceleration voltage should be greater than or equal 20 keV. Very good results have also been archived using 30 keV as an acceleration voltage. In Figure 3 the strategy or mechanism to get an ECCI micrograph of the region of interest is demonstrated. First the sample has to be brought in the right channeling conditions. By zooming out and changing the working distance of the SEM to very high values an electron channeling pattern can be seen. In a slightly zoomed-in image the beam is adjusted to the Kikuchi line of choice where the channeling contrast should be investigated. So called two beam channeling conditions might be favored where the beam is adjusted to the edge where two Kikuchi lines overlap. Only when the beam is adjusted exactly on the edge of the line, like shown in subfigure III, it can be zoomed in (by changing the magnification and the working distance to low values to focus on the surface) to see an ECCI image with sub-micron resolution. To acquire an ECCI image with high quality the scan time has to be rather high. Between 1-5 minutes for a high quality image. Alternatively when the SEM is equipped with the ability to rock its beam, a selected area channeling pattern (SACP), with a much better special resolution can be recorded. This enables a way better adjustment

of the beam to the edge of the Kikuchi lines and further on improves the measurement accuracy and reliability.

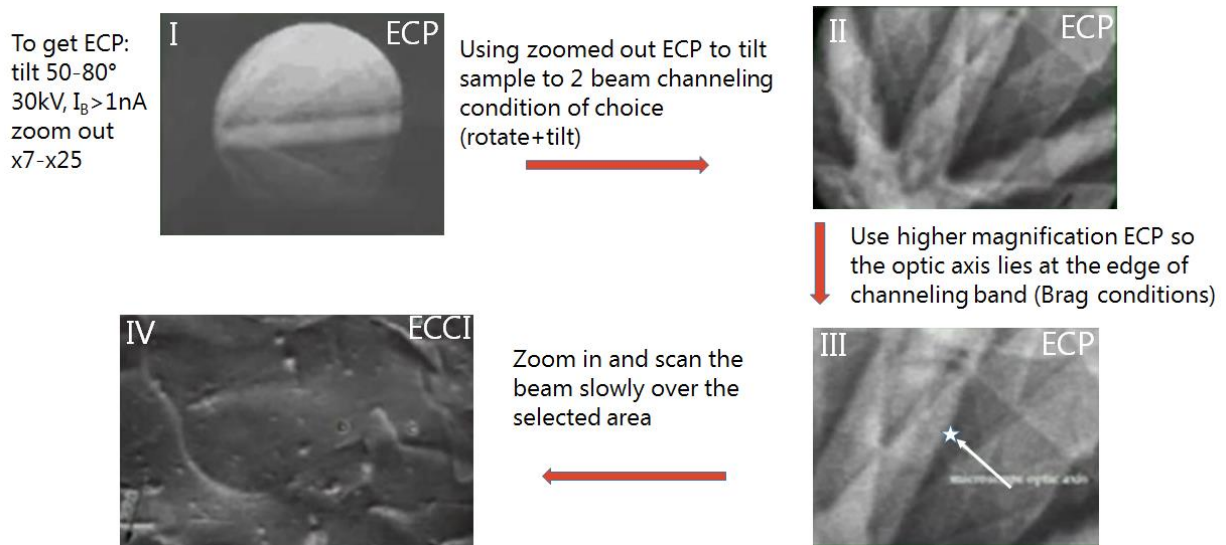


Figure 3: Strategy to successfully perform ECCI measurements. (I) Get the ECP image by zooming out and changing the working distance to very high values. (II) Zoom in into ECP image to get closer to interesting Kikuchi line. (III) Align the center of the screen (marked by the star) exactly to the edge of channeling band. (IV) Zoom in, focus and scan slowly over the selected area to get good quality ECCI images (Images were taken from a talk of M.A. Crimp).

Channeling signal generation

To understand the electron channeling mechanism used to form electron channeling patterns (ECP) and electron channeling contrast images (ECCI) the particle and wave properties of electrons penetrating into the crystal lattice have to be taken into account. In principle the channeling of electrons in some orientations is preferred to others and the electrons can penetrate deeper into the crystal lattice. Some channels/orientations scatter back more electrons than others. This can be used to obtain orientation contrast. The term electron channeling is a little misleading, even though it describes the motion of the electron through the crystal lattice the preferred methods to simulate the channeling patterns in literature use many Bloch wave approaches or other diffraction theories. The channeling contrast can vary due to dislocations in the lattice that increase or reduce the number of backscattered electrons. In Figure 4 the channeling concepts for open and closed channels are shown. The role of dislocations on the backscatter electrons is shown as well as how a crystal lattice channel can be closed by a dislocation.

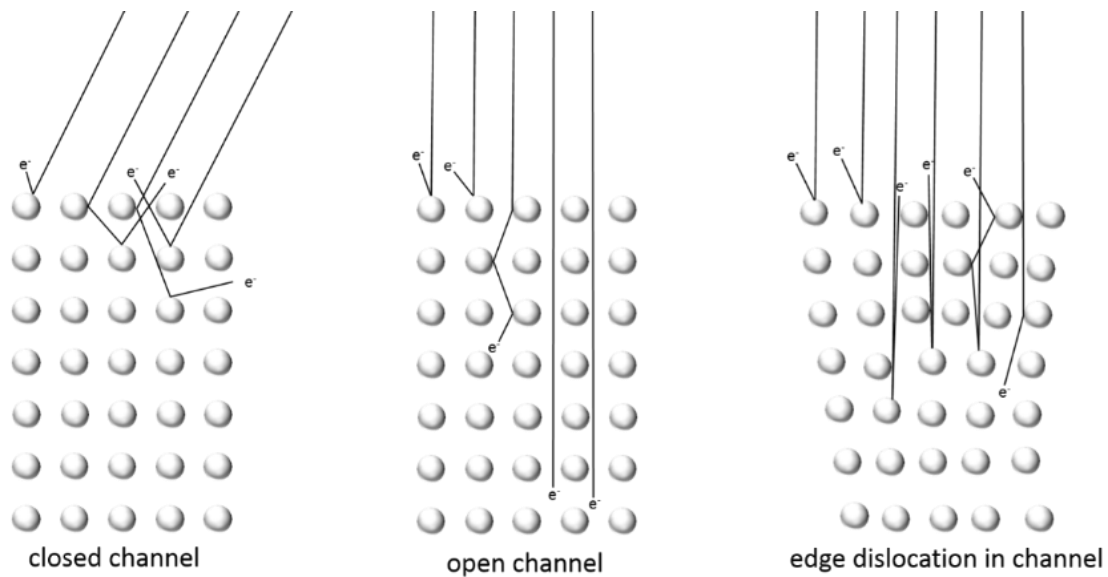


Figure 4: Electron channeling conditions in a SEM when the specimen is oriented in a way so the electrons are confronted with a closed channel (left) where they are stopped close to the surface, or alternatively an open channel (middle) to penetrate deep into the crystal lattice. When an edge dislocation is present and the electrons can penetrate down an open channel (right) the back scattered electrons provide good channeling contrast.

In Figure 5 the backscatter electron signal generation is demonstrated. In most of this work a two-section semiconductor diode back scatter electron (BSE) detector was used. The separate signals detected are S_A and S_B . The separate signals on their own induce a lot of noise. As demonstrated in the figure the sum of the two signals S_A+S_B can be used to visualize compositional contrast and the difference S_A-S_B can be used to show topological contrast. For ECCI measurements the detector is always used in the compositional mode. Still surface features have a strong influence on the channeling signal.

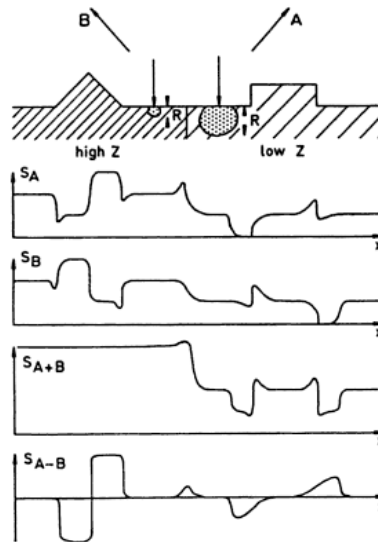


Figure 5: Backscatter electron signal generation in double diode BSE detector in the separate diode detector elements S_A and S_B . Their sum shows best compositional contrast and their difference shows topological contrast.

Applications – Motivation

As mentioned in the history section ECCI measurements have been used for a big variety of applications in the past 30 years, but by far not that extensively like EBIC or TEM investigations. ECCI investigations have been performed on semiconductor materials or metals, especially GaN was a semiconducting material that was investigated several times. The mixed dislocations in GaN are easy to visualize, even if the sample is not oriented in a high symmetry direction. The work of Carnevale et al. (Carnevale, 2014), was the first study of as grown by molecular chemical vapor deposition (MOCVD) materials by ECCI. As shown in Figure 6 (taken from their paper), as-grown (top images) and afterwards annealed (bottom images) specimen with thicknesses from 30-250 nm have been investigated. Due to the penetration depth of the channeling electrons a very good contrast for the misfit dislocations, that are located at the interface, was observed for a layer thickness of 50 nm. For longer distances to the interface, the electrons can't penetrate there anymore and the channeling contrast only shows the threading dislocations at the surface. Nevertheless this study showed that ECCI investigations, can be a rapid non-destructive method to check for dislocations/dislocation densities in materials grown by MOCVD or MBE. For layer thicknesses underneath 50 nm it was not enough channeling contrast to obtain an image. After all, this paper was very pioneering work advertising for the potential of ECCI investigations.

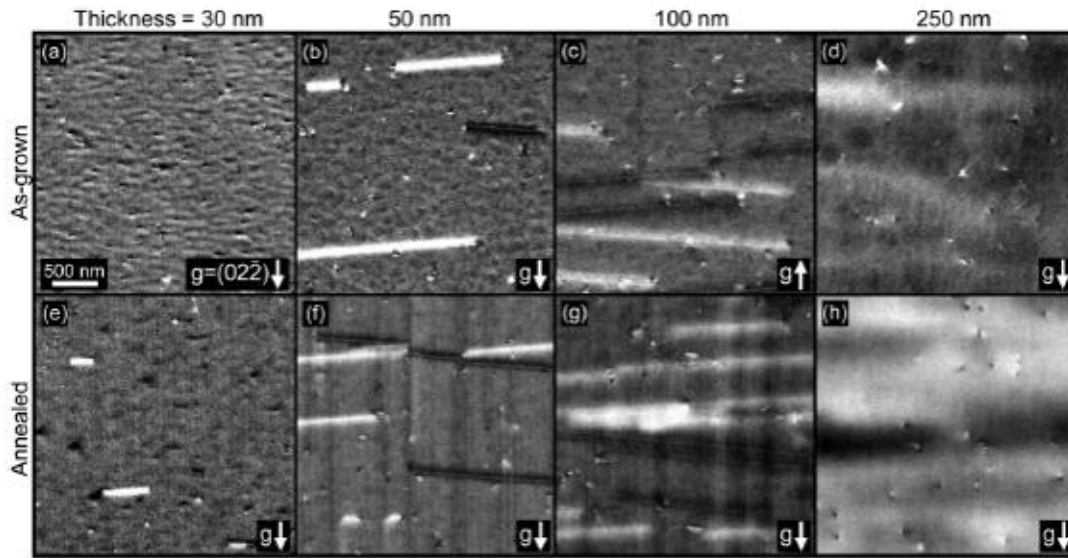


Figure 6: Visualizing of misfit and threading dislocations in as grown (top) and annealed (bottom) GaP layers of different thickness with Electron channeling contrast imaging. Figure taken from (Carnevale, 2014)

In the following table ECCI measurements are compared to other experimental analytical techniques that are used to get information about the TDD in semiconducting materials. The area per picture, the destructiveness, the TDD range and some dis/advantages are pointed out for TEM, EBIC, EPD and ECCI.

| | Transmission Electron Microscope (PVTEM) | Electron Beam Induced Current (EBIC) | Etch Pit Density (EPD) | Electron Channeling Contrast Imaging (ECCI) |
|-------------------|--|---|---|---|
| Area per picture | ~10-100 μm^2 | ~1000-10,000 μm^2 | ~10,000 μm^2 | ~1000-10,000 μm^2 |
| Destructive? | Yes | No | Yes | No |
| TDD range | >10 ⁶ cm ⁻² | 10 ⁴ -10 ⁹ cm ⁻² | <10 ⁸ cm ⁻² | 10 ⁴ -10 ⁹ cm ⁻² |
| Main advantage | Direct imaging of threads | Direct imaging of solar cell areas | Fast and economical | Direct imaging of samples |
| Main disadvantage | Laborious sample preparation | Requires solar cell processing | Requires precise etch depth and smooth surfaces | Requires precise 2-beam condition, indexing ECP |
| Typical images | | | | |

Simulation of Electron Channeling Patterns

As has been mentioned before: to get ECCI images the sample and the detector have to be adjusted in the right orientation. This is normally achieved by tilting and rotating the specimen on the edge of the right channeling line (Kikuchi line) using the electron channeling pattern (ECP) or alternatively an SACP (if beam rocking is possible). To get a better idea what all these lines in the pattern represent and how they are generated, maps like the stereographic map shown in Figure 7 are useful.

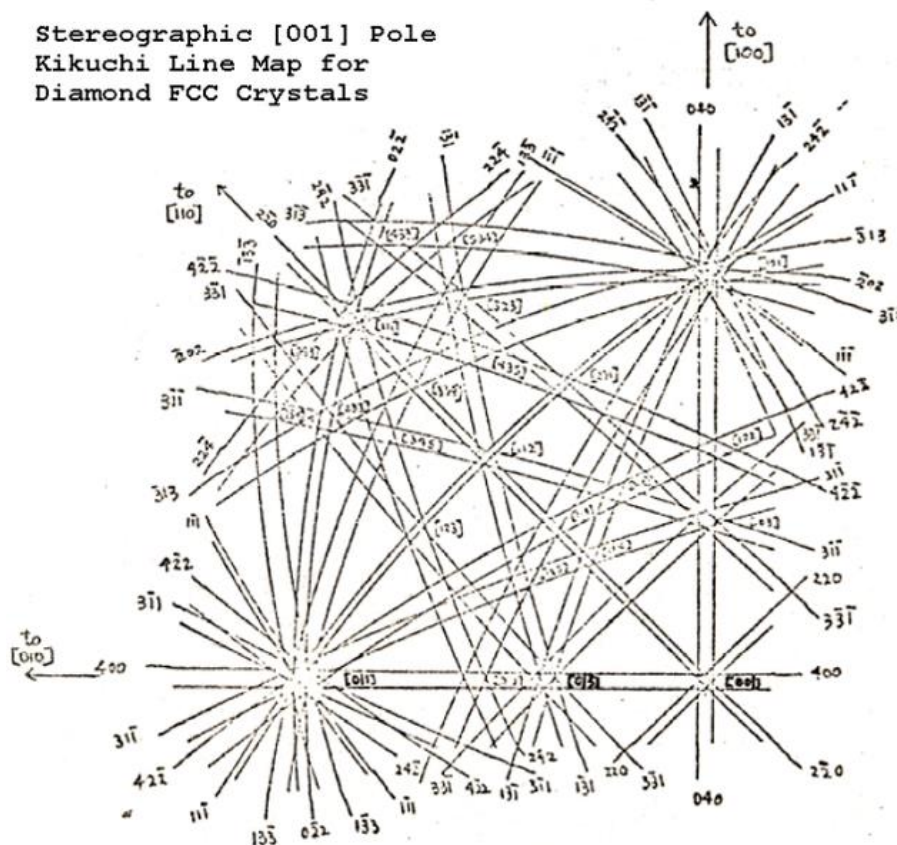


Figure 7: Stereographic [001] Pole Kikuchi Line Map for a diamond fcc crystal structure taken from [Wikipedia.com](https://en.wikipedia.org/wiki/Kikuchi_lines)

Also simple kinematical simulations like shown in Figure 8 can be very helpful, especially in the backscatter geometry where the tilting starts in the middle of the pattern (when the sample is not grown off-cut). When the starting point is not known it can be taff to identify the low symmetry point you are looking at on the ECP pattern (like in forward scatter geometry).

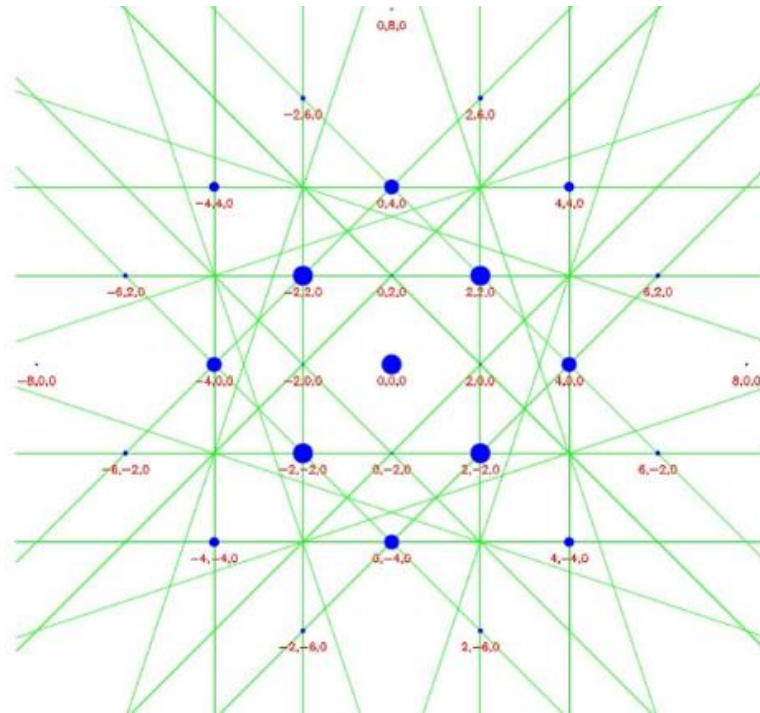


Figure 8: Simple kinematical simulation of Kikuchi line map of GaAsP crystal

In the forward scattering geometry it might happen, that an ECP can be seen but the symmetries are too complicated to indicate the lines. This happened in the very beginning of the project. Prof. S. Zaefferer then simulated the Kikuchi line pattern to our measurement. As can be seen in Figure 9 this made it possible to indicate the high symmetry diffraction lines and to get a better understanding about the generation mechanism of the ECP image generation. Without his help it would have been very hard to identify the lines in the collected electron channeling pattern.

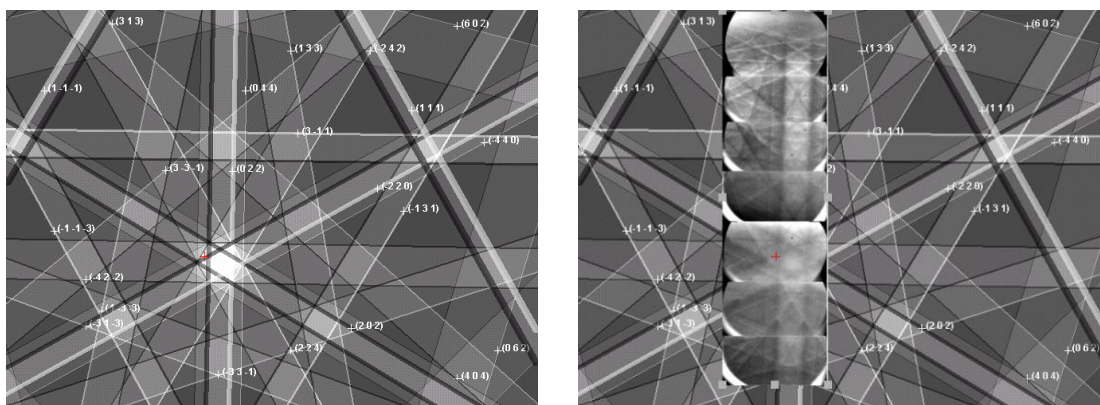


Figure 9: (left) Simulation of ECP image without cropped measurement results (right) simulation and measurement results in one figure.

Electron Beam Induced Current

Electron beam induced current (EBIC) is an analytical technique performed in a scanning electron microscope to visualize built in electric fields or defects in semiconductors. A high energetic electron beam scans over a semiconductor surface, and electrons penetrate into it. Electron-hole pairs are generated locally. These excess charge carriers diffuse around until they recombine with some defect, or when a built in electric field like a pn -junction or a Schottky contact is present, the minority charge carriers might get sucked away and a current can be detected. In Figure 10 the interaction volume of such a high energetic electron beam is demonstrated. As pointed out in the figure, besides EBIC the resulting electrons or x-rays can be used for various purposes.

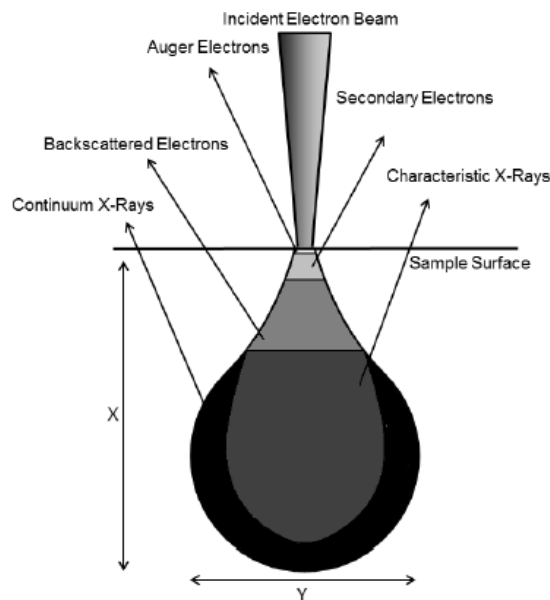


Figure 10: Electron beam interaction volume and extracting electrons and photons/x-rays.

In Figure 11 a typical EBIC line scan over a pn -junction can be seen. The dotted line represents the e-beam direction perpendicular to the pn -junction. As drawn in the back of the image, when the p - and n -regions are homogeneously doped, the maximum of the electric field is located at the metallurgical junction of the two regions. So also the maximum of the EBIC signal is located at this region. Whether the regions are strongly or lightly doped affects the maximum value and the decay of the EBIC signal strongly. Also the presence of defect, that often act as recombination centers can affect the signal strongly.

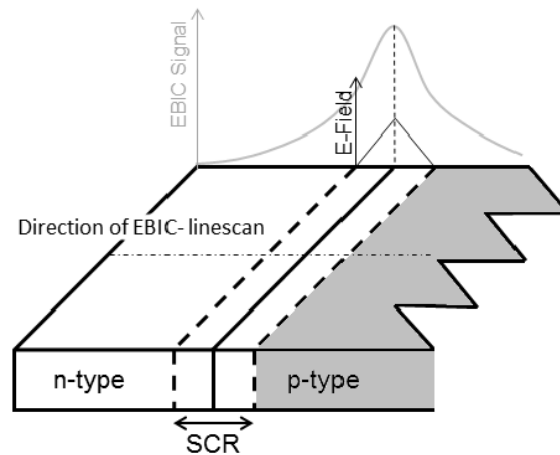


Figure 11: EBIC line scan measurement routine over a pn-junction. The direction of the EBIC measurement, over the SCR of the pn-junction, is shown by the dotted line (figure taken from the master thesis of M. Faccinelli)

In Figure 12 on the left side a secondary electron image and on the right side an EBIC image of the same region are presented. Here a hetero junction between AlGaN and GaN can only be seen as a bright area in the EBIC image. The dark spots on the right side of the junction show threading dislocations. But nevertheless it is obvious that EBIC is a very surface sensitive technique, because surface defect lines from the left to the right side of the sample are also clearly visible. Another drawback of this method is that the resolution limit is dominated by the size of the interaction volume of the electron beam.

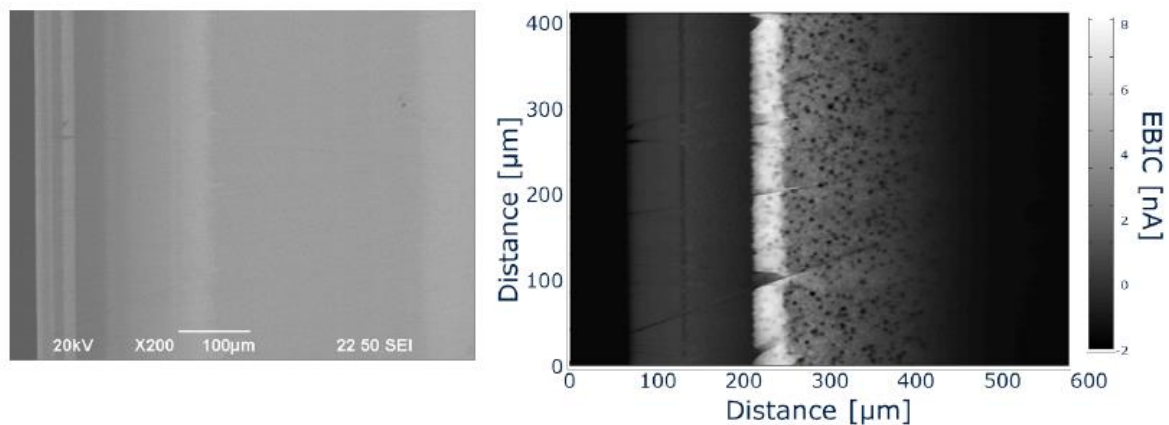


Figure 12: (left) SE image and (right) EBIC measurement of AlGaN/GaN hetero junction. The black points in the GaN region to the right of the junction might be due to an increased concentration of crystal defects (e.g. dislocations) in this layer.

Transmission Electron Microscopy

In comparison to scanning electron microscopy (SEM), where the specimen is irradiated by the electron beam and the electrons interact with the surface of the specimen only, during a transmission electron microscopy (TEM) investigation the electrons from a very high energetic electron beam 200 - 400 keV penetrate through the specimen material. Performing measurements like this resolution limits below 1 Angstrom can be archived. TEM measurement have great potential and the ability to visualize the world of atoms as can be seen in Figure 13. This figure shows a so called platelet defect in proton implanted silicon. As can be seen in the zoomed in image on the right even the atom rows can be seen.

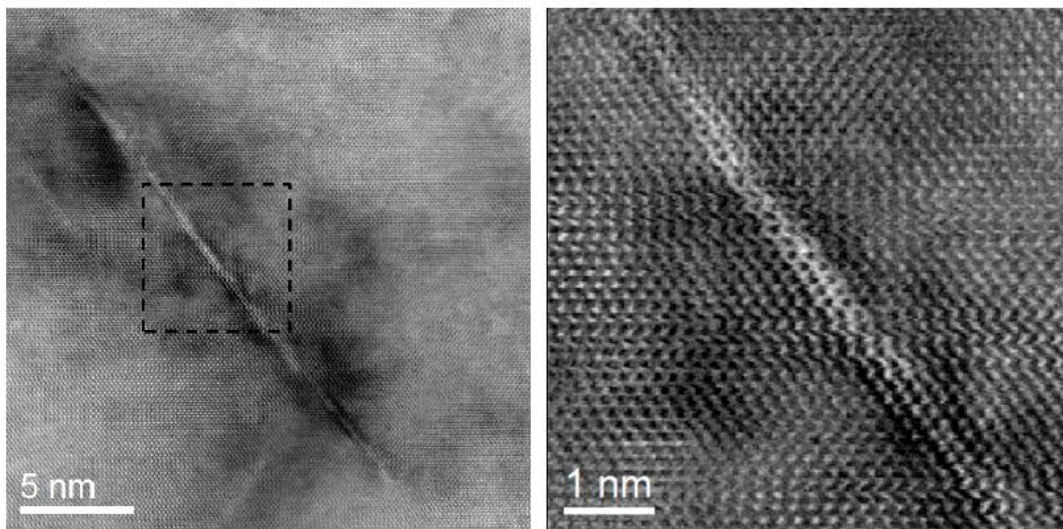


Figure 13: Transmission electron microscopy image of an extended defect in a semiconductor (taken from the PhD thesis of Stefan Kirnstötter)

Besides the great potential of TEM images, the drawback is, that the measurement setup is very expensive, measurement sessions themselves are very time consuming and the sample preparation is also very sophisticated. When samples are thinned to be electron transparent for a TEM measurement, the specimen might lose its bulk character. To get statistically safe/significant values for the dislocation density from TEM measurements is very hard, because the investigated areas are very small and there might be some effect due to the specimen preparation. This has to be taken into account during the analysis section and when the measurement methods are compared with each other.

Additionally to standard TEM investigations, also electron diffraction images can be recorded in a TEM. They can be very useful to investigate the nature of defect complexes detected in semiconductor samples. As can be seen in *Figure 14* from the TEM measurement indirectly diffraction patterns can be generated for different areas. That can be used to investigate differently damaged region it can help to distinguish defects with different crystallographic orientations.

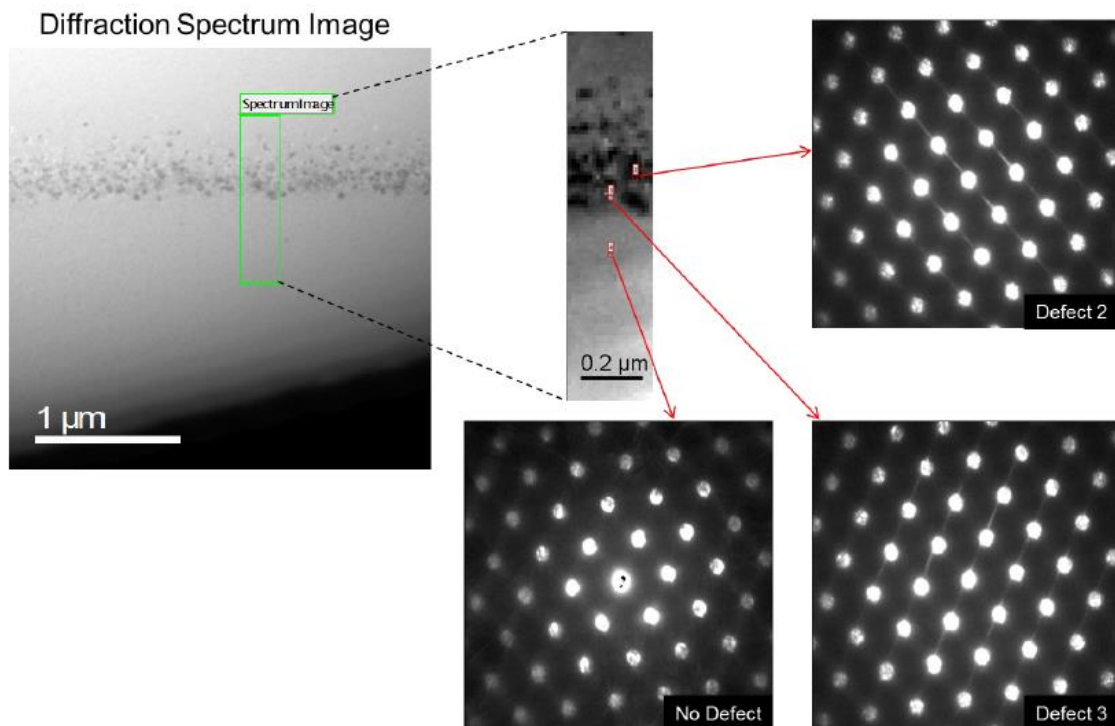


Figure 14: (left) TEM image of proton implanted silicon (middle top) Computed TEM and (left) TEM image of proton implanted silicon. (top, center) Computed TEM and electron diffraction image (right) electron diffraction images of different region around the damaged region formed due to the hydrogen implantation. The red arrows indicate the positions of the electron diffractions (Image taken from the PhD thesis of Stefan Kirnstötter).

Etch pit density

An alternative technique to determine the dislocation density in semiconductor materials is the so called etch pit density (EPD) technique. It can be treated as an alternative for the TDD measurements, and is especially useful for materials without a built in electric field / junction where EBIC cannot be used. Kevin Nay Yaung worked very hard to improve the etching process to be reliable because depending on the III/V semiconducting material the etch rate is very different. In Figure 15 a direct 1:1 comparison of a GaP np-solar cell etched with H_3PO_4 can be seen. In the left image the etch pit density image taken in a Normaski microscope can be seen. The formed pits at the positions of the threading dislocations look like upside down pyramids. In Figure 15 a the etched specimen can be seen. In Figure 15 b and c the EBIC image and the combination of the two images can be seen. Obviously they fit together very well and every etch pit represents a dark spot in EBIC. It should be mentioned that the etching process itself is still not perfectly predictable when different materials like GaAsP instead of GaP are used. Also the resolution limit of this technique is not that low. As can be seen on the 20 μm scale bar, the pits are 5-10 microns wide. When the etching time is reduced to decrease the size of the etch pits, it becomes hard to distinguish the smaller etch pits with surface features/defects that might also act as etching centers in the beginning of the etching process.

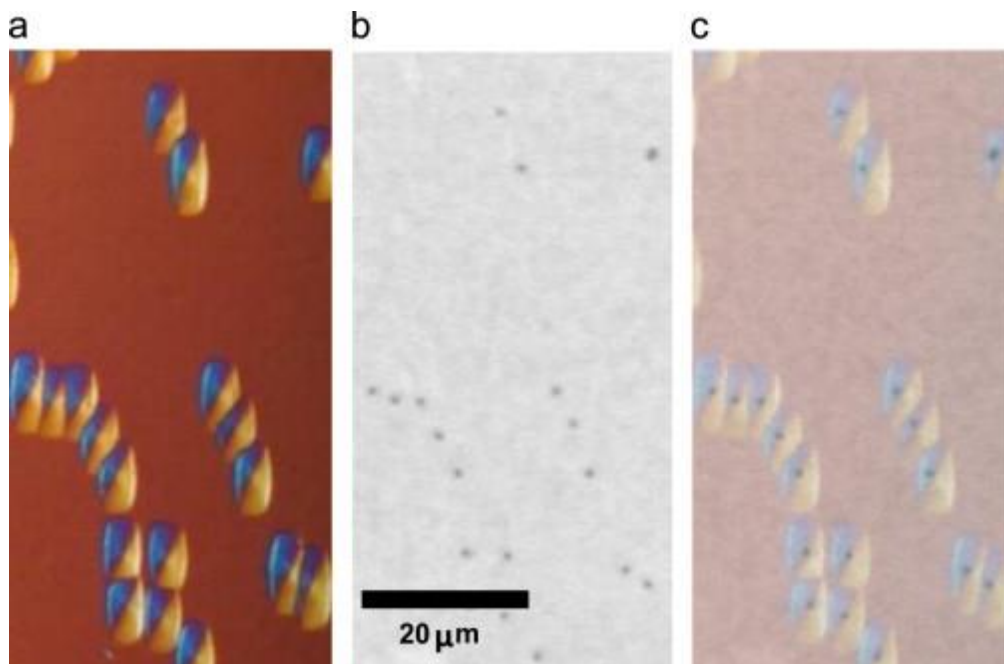


Figure 15: TDD Characterization of a GaP np diode showing (a) Normaski etch pit pattern, (b) EBIC image and (c) EBIC image superimposed on Normaski etch pit pattern. 1-to-1 correlation is shown between the dark spots in EBIC and etch pits obtained by H_3PO_4 DSE.

Chapter II: Experimental Results and Interpretation

Initial Statement

In the experimental results chapter all of the relevant experiments and simulations that were performed in the six months of the research stay at Yale University are presented and interpreted. Most of the studies were performed as a team with Kevin Nay Yaung and some of the results will be published in a journal shortly.

Most of the experimental investigations in the chapter were performed using electron channeling contrast imaging. Lots of different materials, like GaN, GaAs, GaAsP, InAlAs, etc., and device structures like LEDs, solar cells, transistors have been investigated.

Gallium nitride (GaN) – as grown layers and light emitting diodes (LEDs)

The first successful ECCI studies have been performed on GaN. The investigations have been performed in the so called “high tilt” ECCI geometry using two forward scatter diodes to detect the channeling signal. The threading dislocations in GaN are so called mixed dislocations, this means they have different burgers vectors. That results in good channeling contrast also for lower symmetry channeling conditions. In Figure 16 the first ECP and ECCI images on GaN are shown. On the left images the ECP can be seen. The center of the dotted circle represents the position on the edge of a Kikuchi line where the electron beam was focused on. The right images show ECCI images of the very smooth samples. The regular backscatter electron detector signal of the area was just gray and very smooth. So it is clear that the black and with areas represent crystal defects/dislocations.

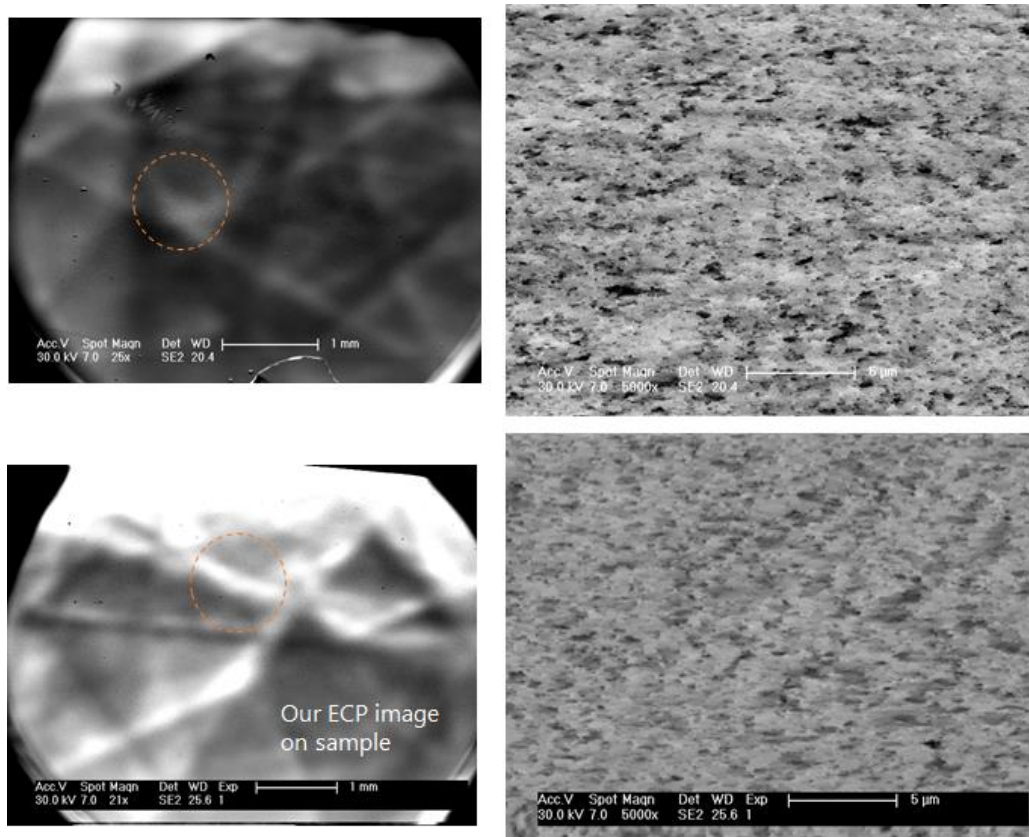


Figure 16: ECP (left) and ECCI (right) images of GaN. The dotted brown line represents the channeling line where the center of the beam was aligned to.

Obviously the contrast was not very good in the ECCI images shown on the right side in Figure 16, this might have been because of the lack of experience of the experimenters. The beam might not have been perfectly aligned to the edge of one channeling line. The magnification should be higher or 10.000x in the beginning, and the detector might not have been mounted close enough to the sample to obtain maximal contrast. Like mentioned in the fundamentals chapter, it is very hard to adjust the ECCI channeling conditions, distance to the detector, distance from the beam to the sample properly in the forward scattering geometry.

Nevertheless, when these points can be improved, good dislocation contrast, comparable to results presented in literature can be obtained. As can be seen in Figure 17 the ECP image is way finer and shows more details due to the closer forward scatter diode detector. Also the dislocations can be visualized as focused dark/bright contrast features. Unfortunately during the measurement the specimen was not properly electrically contacted to avoid charging and therefore the beam was affected by the deposited charges on the surface during the measurement. That's why some parts of the right image look slightly shifted.

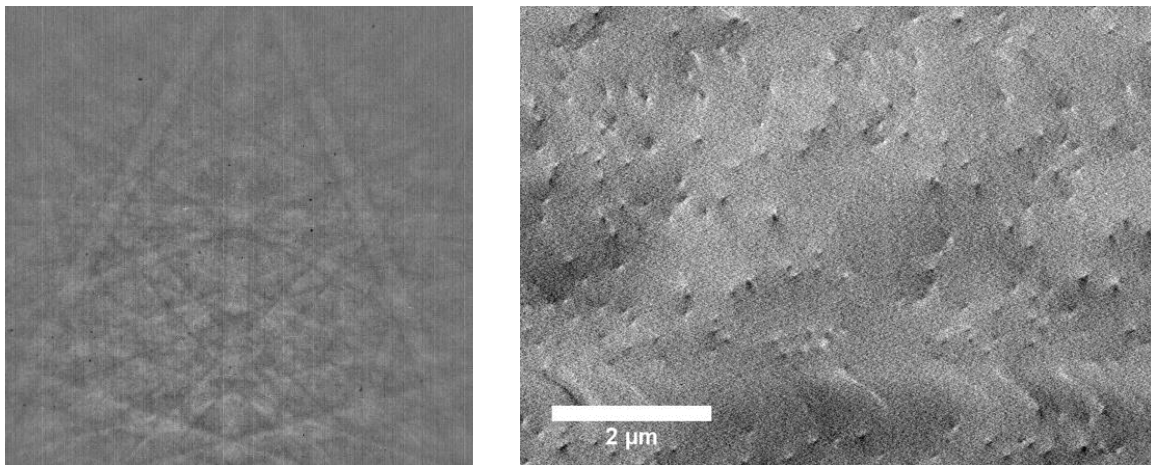


Figure 17: ECP (left) and ECCI (right) image of an area on a GaN-LED

Quantum dot (QD) and quantum well (QW) laser structures

Various laser structures with $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ QD or QW layers and GaAs top layers have been investigated using TEM, EBIC and ECCI measurements. On some of the specimens EBIC measurements have not been possible, due to the weak processing (metal deposition), small built in electric field and bad electrical contacts. One of the very common growth structures is shown in Figure 18. The laser structure was grown on GaAs, GaP and GaP/Si substrates via molecular beam epitaxy.

| |
|--|
| 100 nm p^+ -GaAs contact layer, 510 °C |
| 1 μm $\text{p-Al}_{0.3}\text{Ga}_{0.7}\text{As}$, 690 °C |
| UID- $\text{Al}_x\text{Ga}_{1-x}\text{As}$ GRIN layers |
| 8 nm UID- $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ QW, 530 °C |
| UID- $\text{Al}_x\text{Ga}_{1-x}\text{As}$ GRIN layers |
| 1 μm $\text{n-Al}_{0.3}\text{Ga}_{0.7}\text{As}$, 690 °C |
| 200nm n-GaAs , 630 °C |
| 2 μm n^+ -GaAs, current spreading layer, 630 °C |
| 100 nm n-GaAs , 500 °C |
| a. GaAs, b. GaP substrate, c. GaP/Si (001) |

Figure 18: Quantum-Well Laser Structure (figure taken from Xue Huang)

The morphology of the top GaAs layer was investigated with atomic force microscopy. The images in Figure 19 clearly show the differences in the surface roughness. When the stack was grown on GaAs the top layer is very smooth and much rougher when grown on GaP or GaP/Si. As can be seen in the following sections, the roughness of the surface is a good indicator for the threading dislocation density.

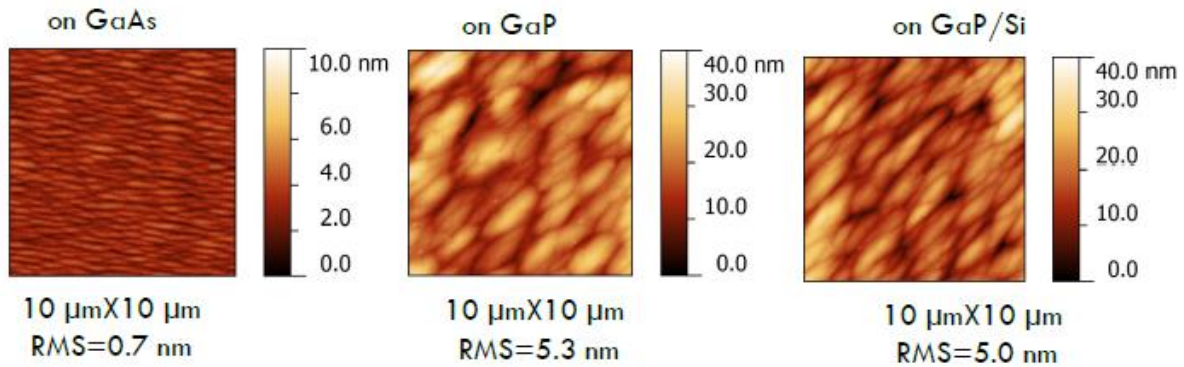


Figure 19: Atomic force micrograph of laser structures grown on GaAs (left), GaP (middle) and GaP/Si (right)

All ECCI investigations were only performed of the top layer. Theoretically it would have been possible to perform a measurement after every growth step. This is planned for the future but was not done for this set of samples. In samples grown on GaAs the TDD is way below $1 \times 10^6 \text{ cm}^{-2}$ and can't be visualized with ECCI to get a statistically significant value. So in Figure 20 the laser structures grown on GaP (left) and GaP/Si (right) investigated with ECCI are shown. As can be seen in the images the threading dislocation density is about a factor 1.5-2 higher in the specimens grown on GaP than on the specimens grown on GaP/Si. This is not a big difference in comparison to the laser structures grown on GaAs with a TDD $< 1 \times 10^6 \text{ cm}^{-2}$. Nevertheless it is very consistent for this three sets of structures. In the three different samples, the growth conditions (growth temperature and growth rate) have been changed and the samples with the same set of growth parameters are compared in this image.

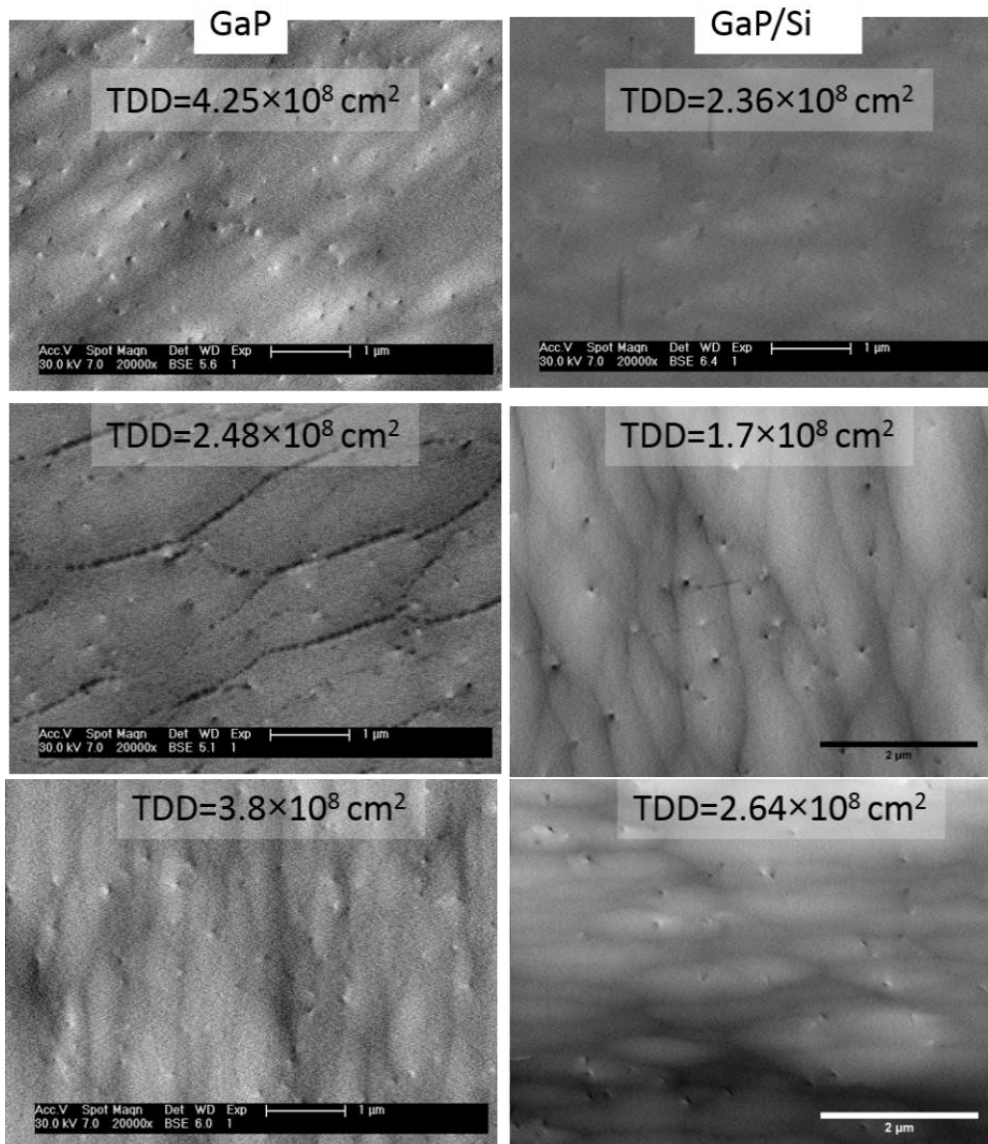
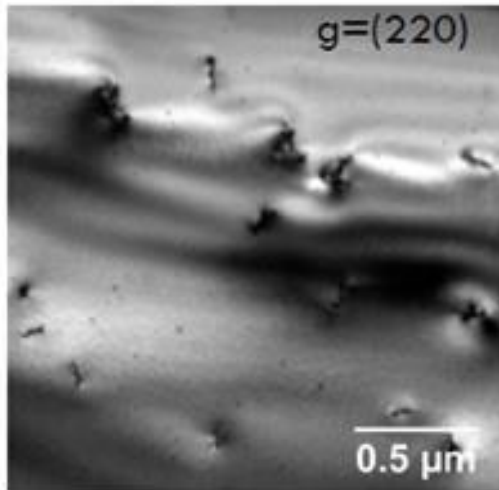


Figure 20: ECI investigation of GaAs top layer of laser structure grown on GaP (left) or GaP/Si (right) substrate.

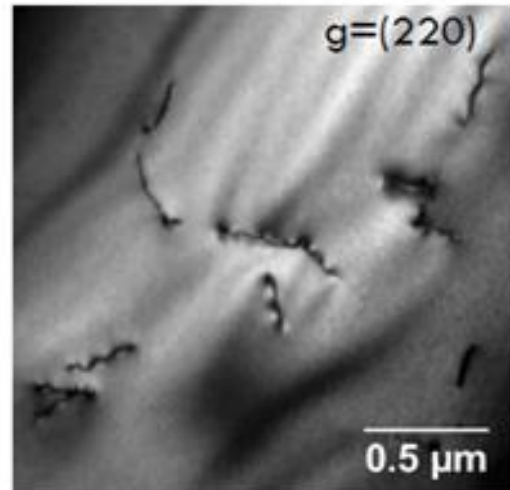
To get statistically significant values for the TDD for the different specimens, about 3-5 images in various channeling conditions (e.g. (220), (400)) have been recorded and analyzed. In Figure 21 TEM investigations in (220) orientation are presented. Here 15 areas of $2.8 \times 2.8 \mu\text{m}^2$ have been investigated to get statistically significant values for the TDD. Nevertheless the results was about a factor of 2 smaller than the values obtained from ECI. Also on structures grown on GaP/Si the TDD is smaller.

15 consecutive PVTEM images ($2.8 \times 2.8 \mu\text{m}^2$) for TDD count

$$\text{TDD}_{(\text{GaP})} \sim 2.6 \times 10^8 \text{ cm}^{-2}; \text{TDD}_{(\text{GaP}/\text{Si})} \sim 1.3 \times 10^8 \text{ cm}^{-2}$$



PVTEM of GaP sample
TDD $\sim 2.6 \times 10^8 \text{ cm}^{-2}$



PVTEM of GaP/Si sample
TDD $\sim 1.3 \times 10^8 \text{ cm}^{-2}$

Figure 21: TEM investigation of threading dislocations in GaAs top layer in QW-laser structure grown on a GaP (left) or a GaP/Si (right) substrate.

A discrepancy between the measurement results in TEM and ECCI like this, is difficult to analyze and interpret. Obviously there is a systematic error because the trend seems to be the same in all of the measurements. It might be that the experimenter is kind of biased to go to regions on the surface with dislocations in it – to focus properly. That might result in a higher value for the TDD than it is on average over the whole sample. The TEM measurements have not been performed by the same persons who did the ECCI investigations, what could affect it somehow. The burgers vector for the imaging conditions in TEM was $\vec{g} = (220)$, in ECCI this was not that clear. It could also have been a $\vec{g} = (220), (\bar{2}\bar{2}0), \dots$ This might have had an influence on the channeling conditions in ECCI.

Misfit dislocation investigations – GaAs on GaP/Si

Besides threading dislocation that grow from the interface where they are generated to the surface, misfit dislocations are stuck at the interface. Nevertheless these defects are important for the material quality and later device performance too. In this section a study of GaAs solar cells on GaP/Si is presented. The misfit dislocations in the solar cells are investigated using EBIC and ECCI measurements. In Figure 22 EBIC and ECCI images of a region on a GaAs solar cell grown on GaP/Si are shown. Obviously in the secondary electron (SE) image no misfit dislocations can be seen. The top GaAs layer has a thickness below 50 nm nevertheless, secondary electrons only provide signals from the surface and maybe the first few nanometers. For EBIC this is the perfect depth (the junction is a few hundred nm underneath misfit dislocations), so the diffusing excess charge carriers can recombine at the misfit dislocations and provide a clear signal (as can be seen in the EBIC image top left).

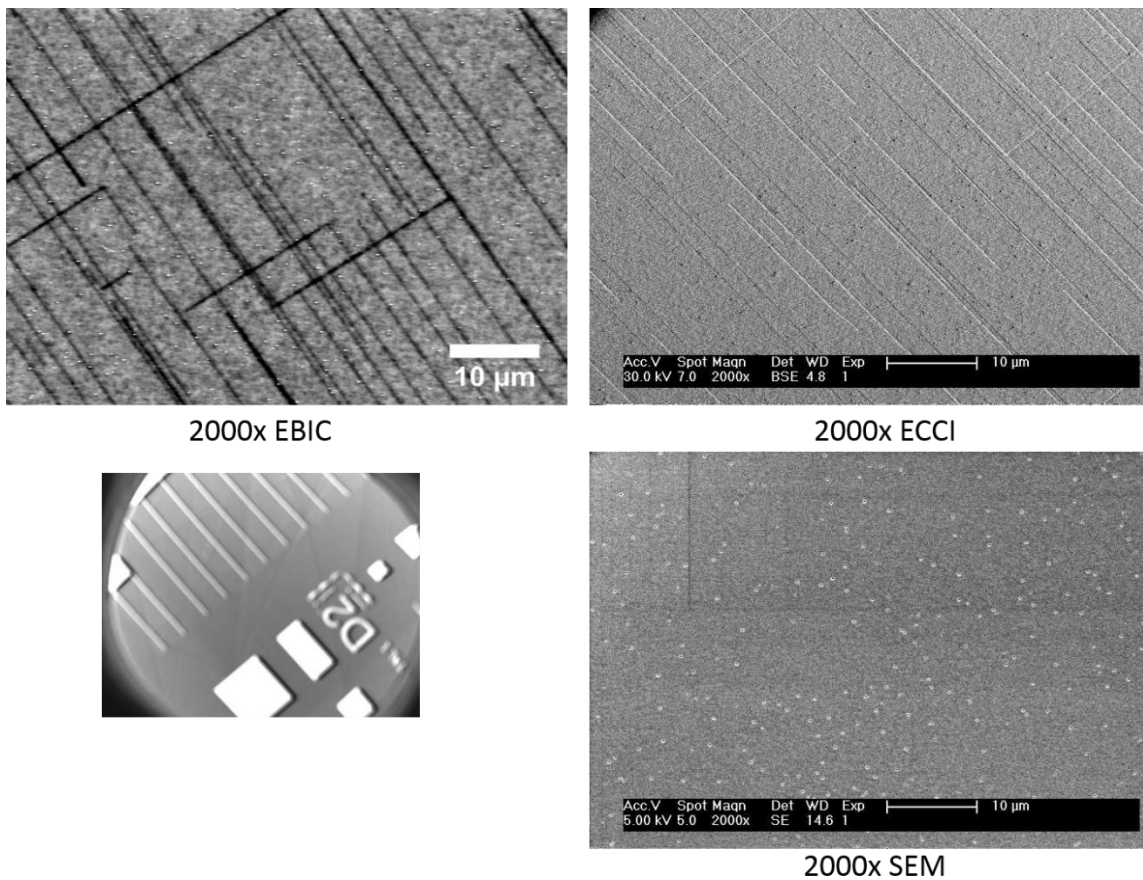


Figure 22: (top left) EBIC, (top right) ECCI, (bottom left) ECP and (bottom right) SE image of GaAs solar cell grown on GaP/Si.

As can be seen, the misfit dislocations show up at the same positions in EBIC and ECCI. As can be seen in Figure 23 the misfit dislocations can be seen clearly when the electron beam is adjusted to the (400) channeling band, as can be seen in the top right edge of Figure 23. When the beam was adjusted to the (260), a lower symmetry line, no misfit dislocation contrast was visible in the ECCI image any more. This might have to do with the burgers vector of the misfit dislocations and needs further investigation.

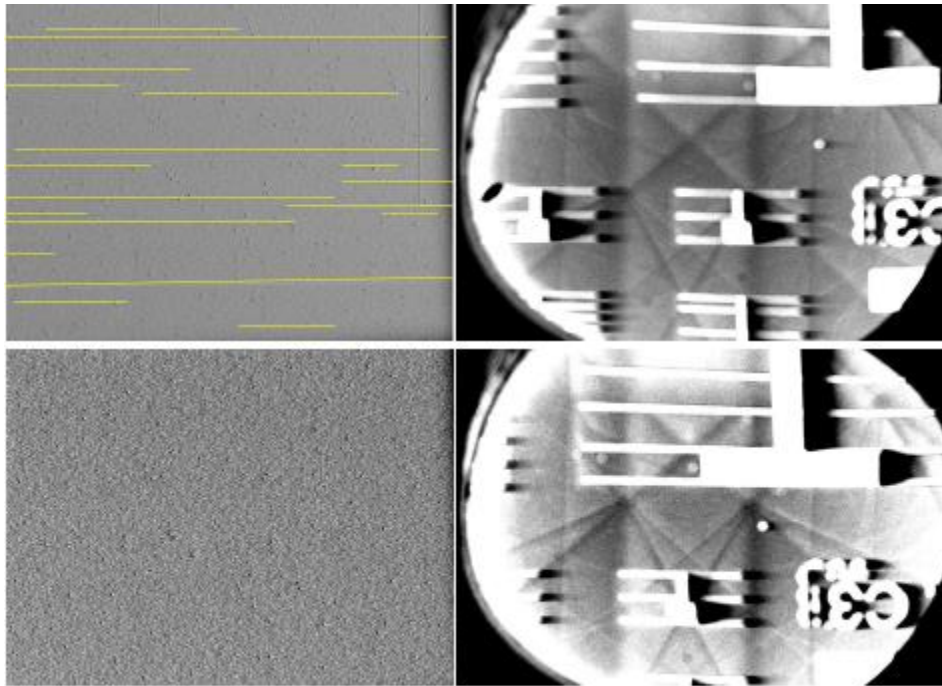


Figure 23: ECCI (left) and ECP (right) images of GaAs solar cell. Misfit dislocations are highlighted with yellow color. A vertical misfit line was not highlighted to show its shape in the top left image. As can be seen in the top ECP the ECCI to the left was recorded in (400) channeling condition. The bottom images were adjusted to (260) channeling conditions and no misfit line was visible in ECCI.

When the specimen was adjusted to higher symmetry channeling conditions, like shown in Figure 24. Here the electron beam was pointed at the edge of a (220) line in the ECP pattern and a very clear contrast for the misfit lines was archived. When the beam was then adjusted to another channeling line of the (220) family, the vertical misfit line vanished in the bottom ECCI image.

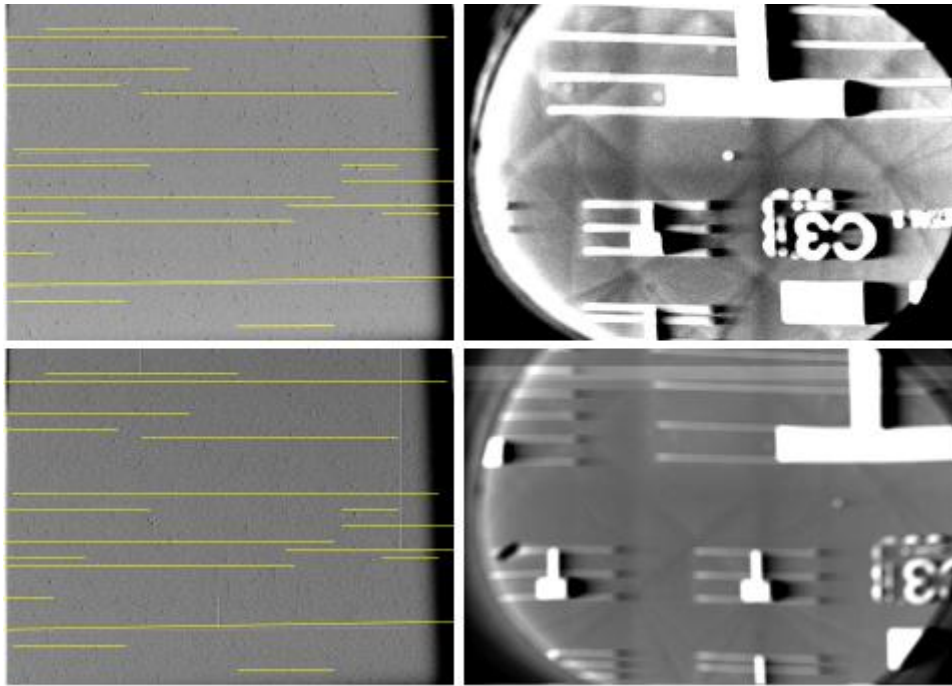


Figure 24: ECCI (left) and ECP (right) images in (220) channeling condition of GaAs solar cell. Misfit dislocations are highlighted with yellow color.

InAlAs graded buffer layers

In this section, InAlAs graded buffers have been investigated. These different variations of intermediate buffer layers were used to improve the material quality of the top layer. They were grown in several steps and the growth ratio of the components In, Al and As were slightly changed during the process. As can be seen in Figure 25 the InAlAs graded buffer layers provided a very clear contrast in the ECP image, therefore it was easy to focus on the edges of the right channeling lines.

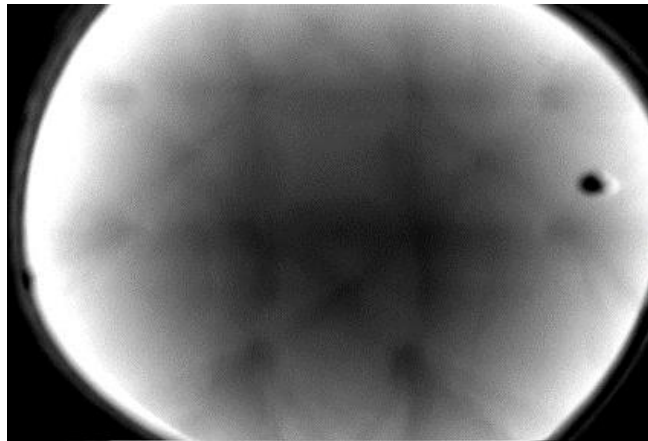


Figure 25: ECP image of an InAlAs graded buffer.

Both graded buffer layers improved the surface quality of the top layer in comparison to the sample without a buffer layer. But as can be seen in Figure 26 **Fehler! Verweisquelle konnte nicht gefunden werden.** and Figure 27 in the backscatter electron images of both layers, the surface morphology of the graded buffer layer 1 (Figure 26) is finer than graded buffer layer 2 (Figure 27). The backscatter electron image was taken with the same detector as the ECCI images have been taken with. But, during the regular measurement the sample was not oriented close to any channeling condition. Additionally the detector-diodes were in the A-B mode instead of the A+B mode in ECCI, to generate the best topological signal. It can be seen that the morphology influences the density of threading dislocations, the TDD is about 30% higher in the top layer grown on the graded buffer 2.

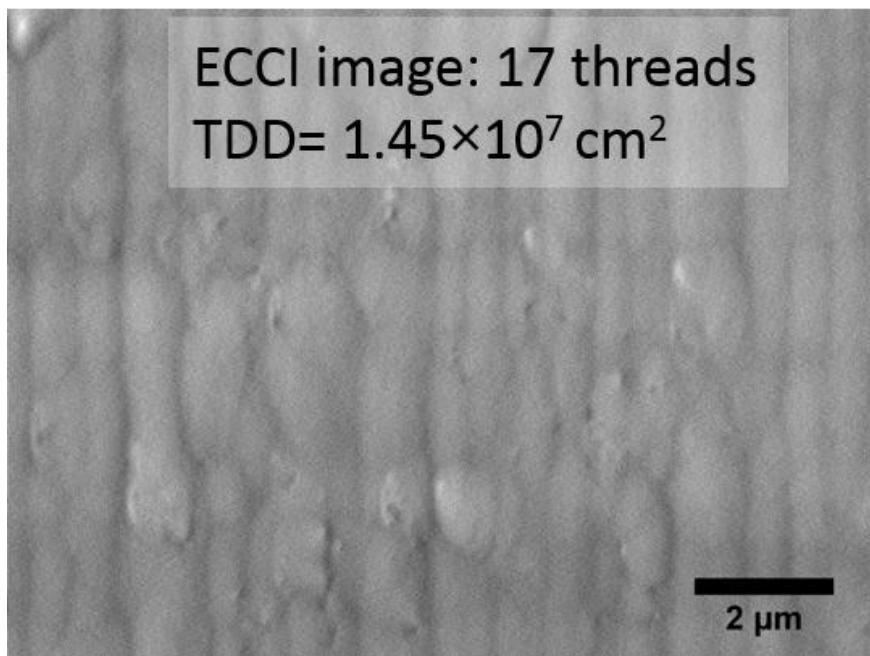
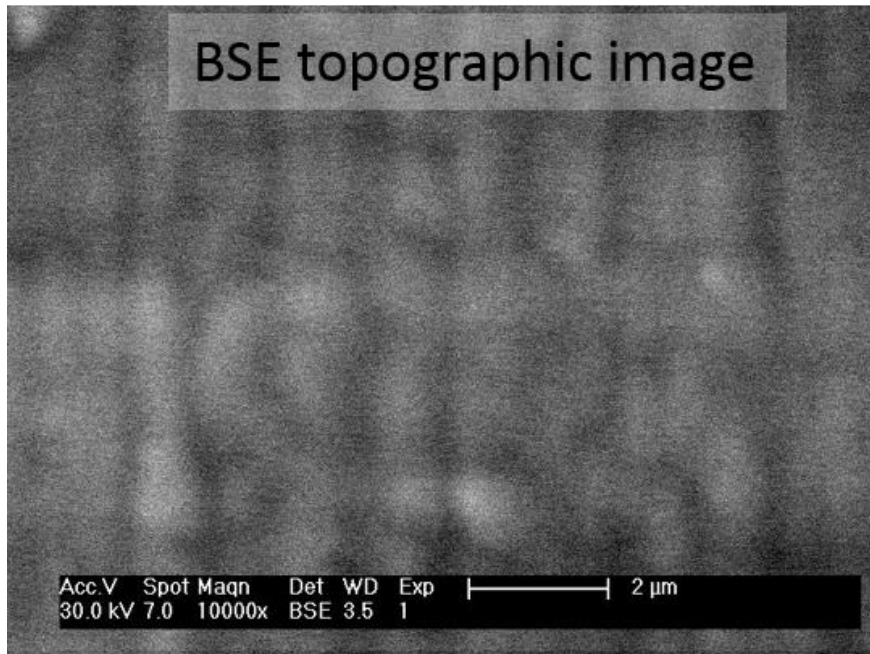
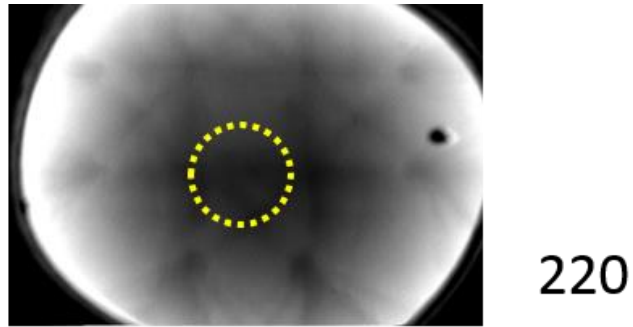
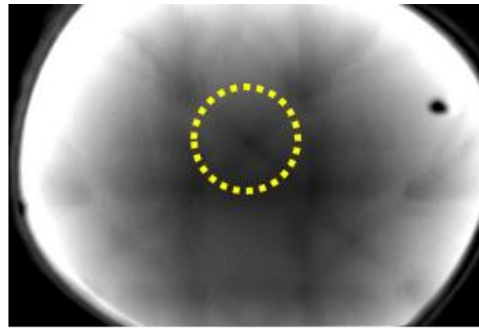


Figure 26: ECP (top), topographic back scatter electron (BSE) (middle) and ECCI image (bottom) of graded buffer layer structure1. The specimen was adjusted in the 220 channeling conditions.



400

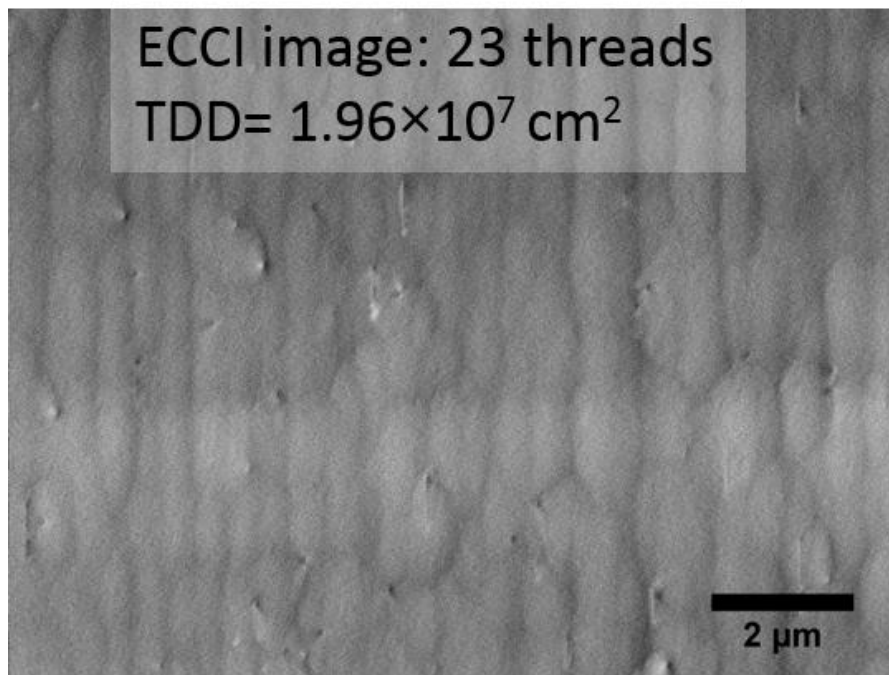
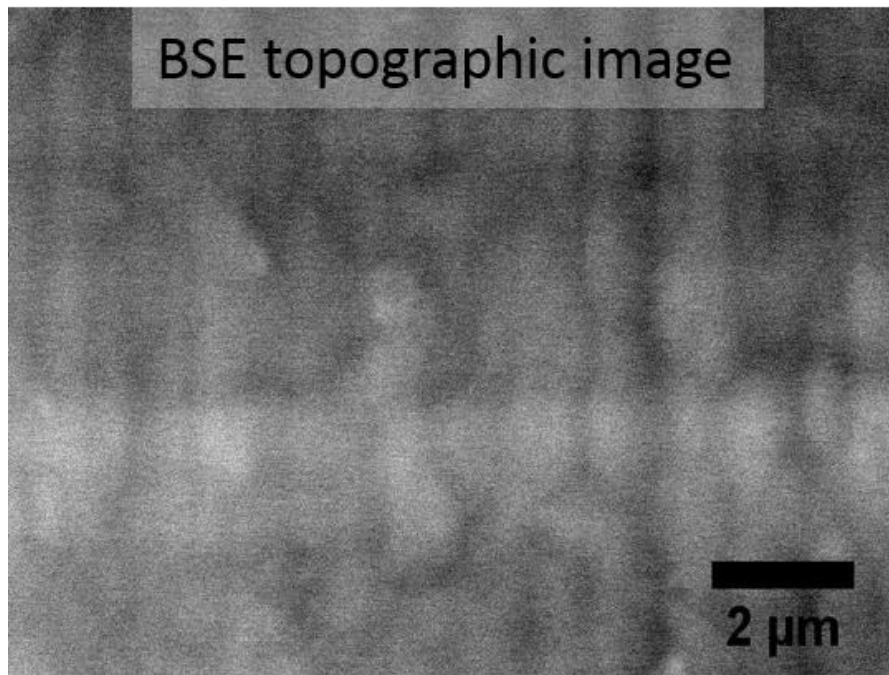


Figure 27: ECP (top), back scatter electron (BSE) (middle) and ECCI image (bottom) of graded buffer layer structure 2. The specimen was adjusted in the 400 channeling conditions.

Direct 1-to-1 EBIC-ECCI comparison

The direct 1-to-1 comparison of analytical techniques is very important when a new method should be added to a repertoire of tools for the analysis. In this section the comparison of EBIC and ECCI investigations is discussed and some measurement results are presented. EBIC is an important technique in the analysis of defects in solar cells and diodes. EBIC measurements can be performed when the semiconducting sample has a built in electric field, like a *pn*-junction or a Schottky contact. ECCI measurements can also be performed without a built in electric field present. Nevertheless several additional conditions have to be fulfilled: the surface has to be very clean and $TDD \geq 1 \times 10^6$. Even though EBIC is also very surface sensitive, if surface defects are present that are about the size of dislocation contrast features it is nearly impossible to generate a reliable ECCI image. The first comparison that was recorded is shown in Figure 28.

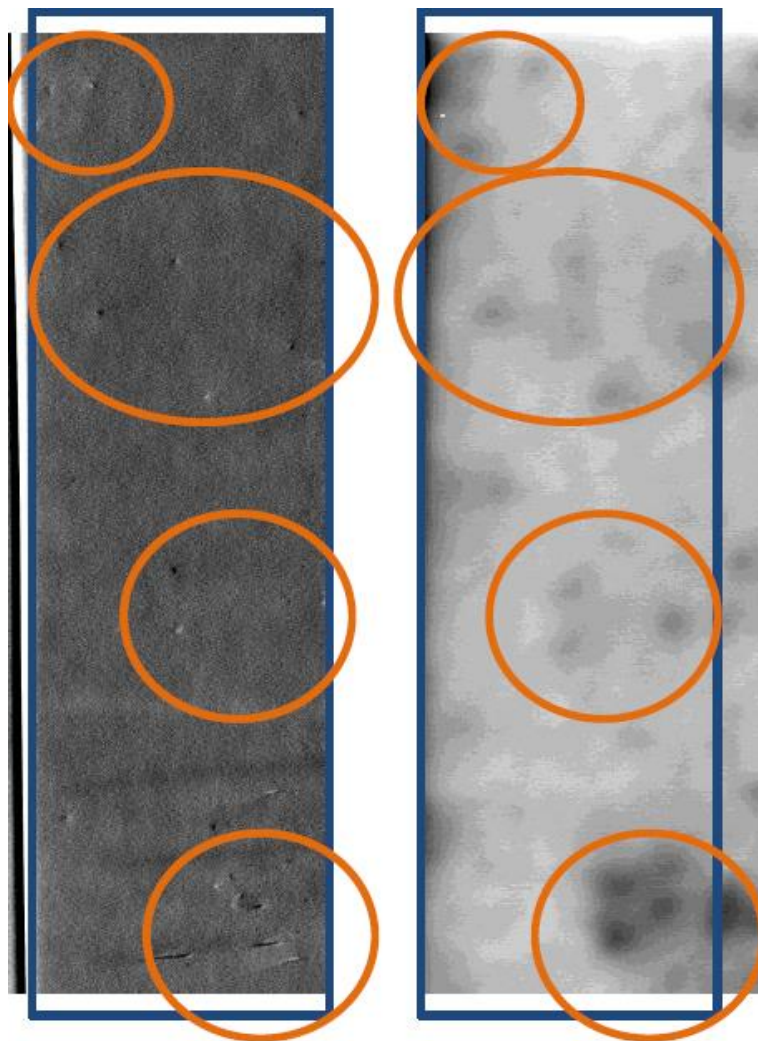


Figure 28: Direct 1-to-1 ECCI (left) and EBIC (right) comparison.

The orange circles point out region with defects present. Obviously the threading dislocations have a very narrow sharp black-white contrast using ECCI and are a little blurred out using EBIC.

As can be seen in Figure 29 in another 1-to-1 correlation study of an area investigated with ECCI and EBIC, there are sometimes defects / dislocations present in an image taken with one technique but not with the other. In the bottom of Figure 29 the ECCI image shows no dislocation where clearly a dark area is present in the EBIC image.

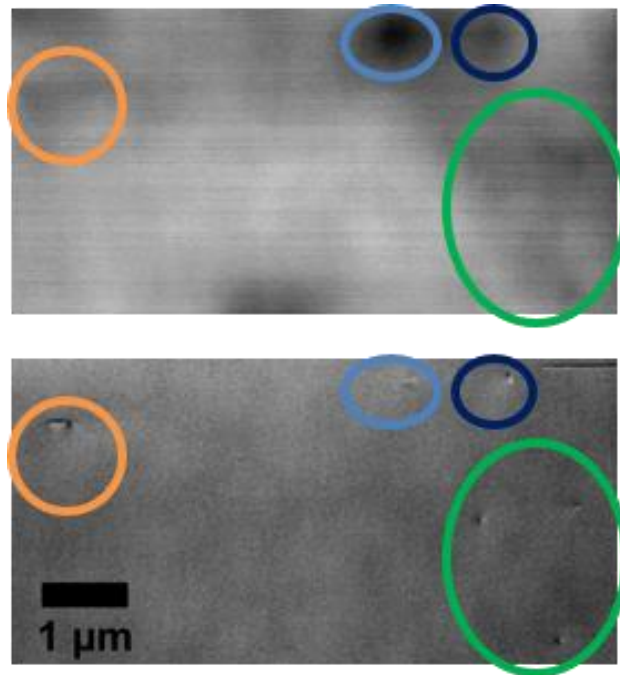


Figure 29: GaAsP/SiGe solar cell: (top) 10,000x EBIC image (bottom) 10,000x ECCI image. Close 1-to-1 correlation of up to 75% is shown.

After all, an about 75-85% correlation between the defect densities detected in both methods was recorded. Some surface defects are very dominant in ECCI and some recombination effects in EBIC might also affect the results of the analysis. Nevertheless the absolute number of the defects is comparable.

Direct 1-to-1 EPD-ECCI comparison

In this section a 1-to-1 etch pit density (EPD) and electron channeling contrast imaging (ECCI) comparison study is shown. Like in the previous EBIC-ECCI comparison the results are not perfectly the same when analyzed with different techniques. As can be seen in Figure 30 the dislocations can be visualized with both techniques. The ECCI measurements have been performed prior to the etching process. The sample was arranged in the (220) channeling conditions for the ECCI investigations. The etching process was performed like explained in the fundamentals section. It was optimized to obtain clear and as small as possible etch pits. Otherwise, when the etch pits would have their regular size of about 4-8 μm , it would have been difficult to compare the areas where a lot of dislocations are agglomerated.

As can be seen in Figure 30, again a 1-to-1 correlation of about 75% of the defects was detected. This discrepancy can be because of some problems during the ECCI investigation like mentioned above (surface sensitive method, sometimes it's hard to judge if a feature is dirt or a dislocation), or some problems during the etching process. When the sample is not etched long enough, some surface defects (dirt, scratches,..) can be decorated and etched and might look like an etched dislocation. When the etching process is continued to an etch pit size of about 3-5 μm it is very clear what represents a dislocation and what was just etched slightly.

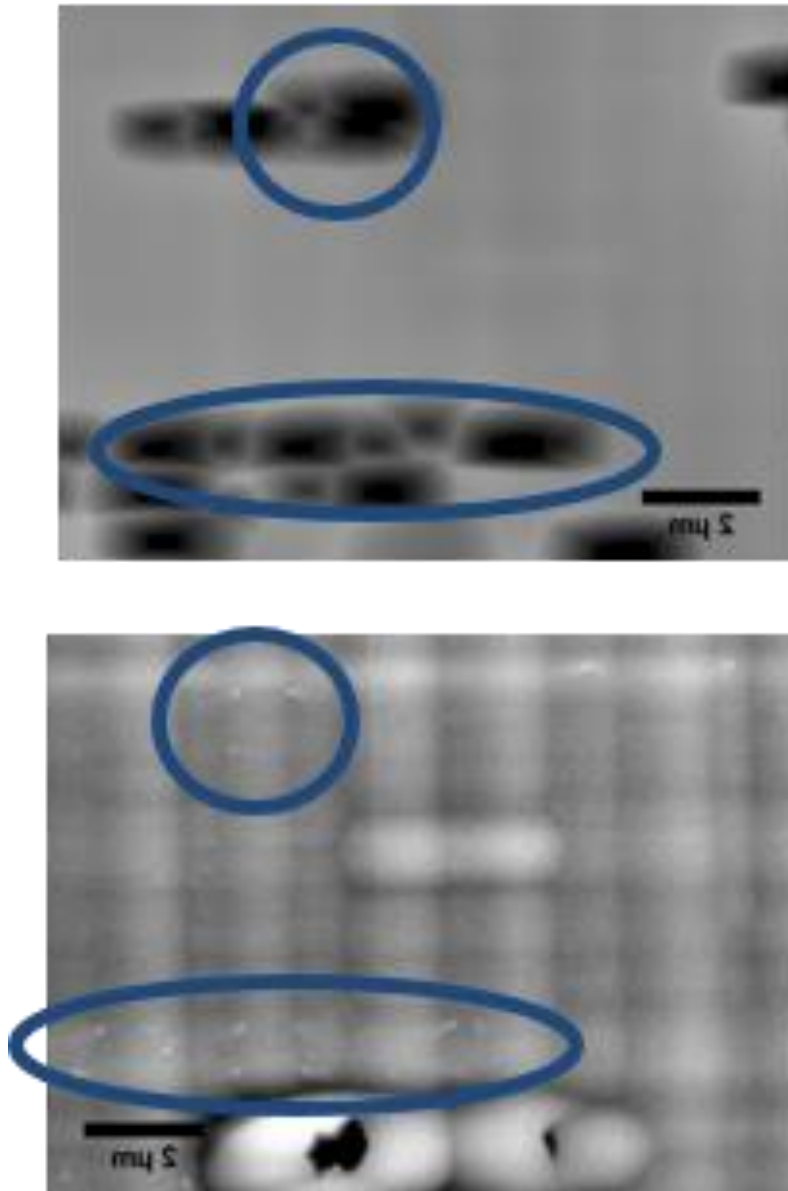


Figure 30: Direct 1-to-1 EPD (top) and ECCI (bottom) comparison. The blue circles imply the areas with a high number of defects. Scale bar represents 2 μ m.

Anti-phase domains

Besides dislocations and stacking faults, anti-phase domains (APD) are a very prominent type of defect present in semiconductors. Like demonstrated in Figure 31 an anti-phase domain is an area where the atoms are arranged in the opposite order then in the otherwise perfect crystal lattice. In the figure this is shown by the different regions A and B. In B the Ga and As atoms are upside down in comparison to the perfect crystal lattice in region A.

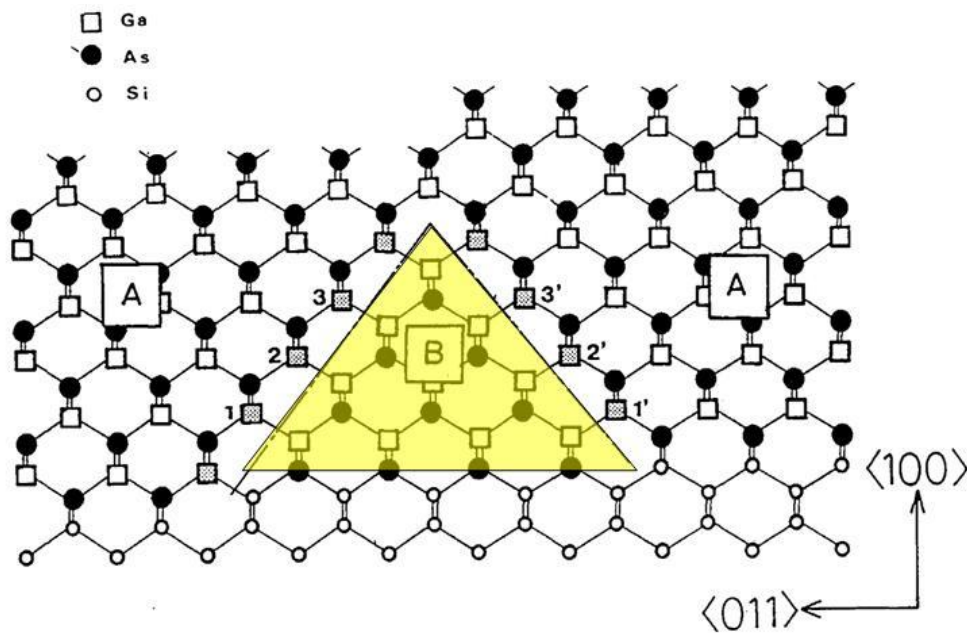


Figure 31: Schematical visualization of an anti-phase domain. Figure taken from Wikipedia.com.

A sample with a lot of APDs was investigated with EBIC and ECCI and as shown in Figure 32. The general shape of the APD look the same no matter which analytical technique is used. Obviously in the EBIC images the lines around the anti-phase domain are not that fine. Some dark area can be recognized in the center of the domain and multiply dark areas around the domain boundaries in the EBIC image. In the ECCI images these dislocations can be seen much sharper and it is obvious that there are multiple dislocations present in the APD. This again points out, the superior resolution limit of ECCI that is very useful to analyze agglomerations of dislocations.

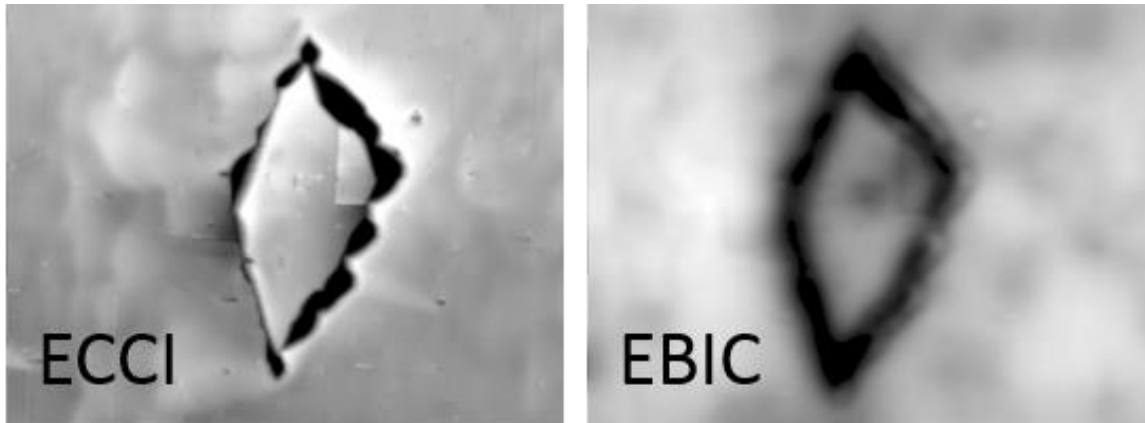


Figure 32: (left) ECCI and (right) EBIC image of an anti-phase domain.

ECCL-studies over several orders of magnitude of the TDD

In this section the usability of ECCL over several orders of magnitude of the TDD and the statistical reliability of the measurements will be demonstrated and discussed. As can be seen in Figure 33 ECCL studies can be performed over two orders of magnitude from the low 10^6 to the medium 10^8 threading dislocations per cm^2 . When the TDD is below 10^6 cm^{-2} it is hard to find dislocations with ECCL because, to get optimal results, the measurements have to be performed at a magnification of $\times 10,000$. A TDD below 10^6 represents a little less than one dislocation in this area on average. Then it is very hard to distinguish dislocations from surface defects and to bring the sample in the correct channeling conditions. When the TDD is high it is very obvious when the sample is tilted and gets into the (220) channeling condition, because a lot of dislocations pop up and become visible in the image.

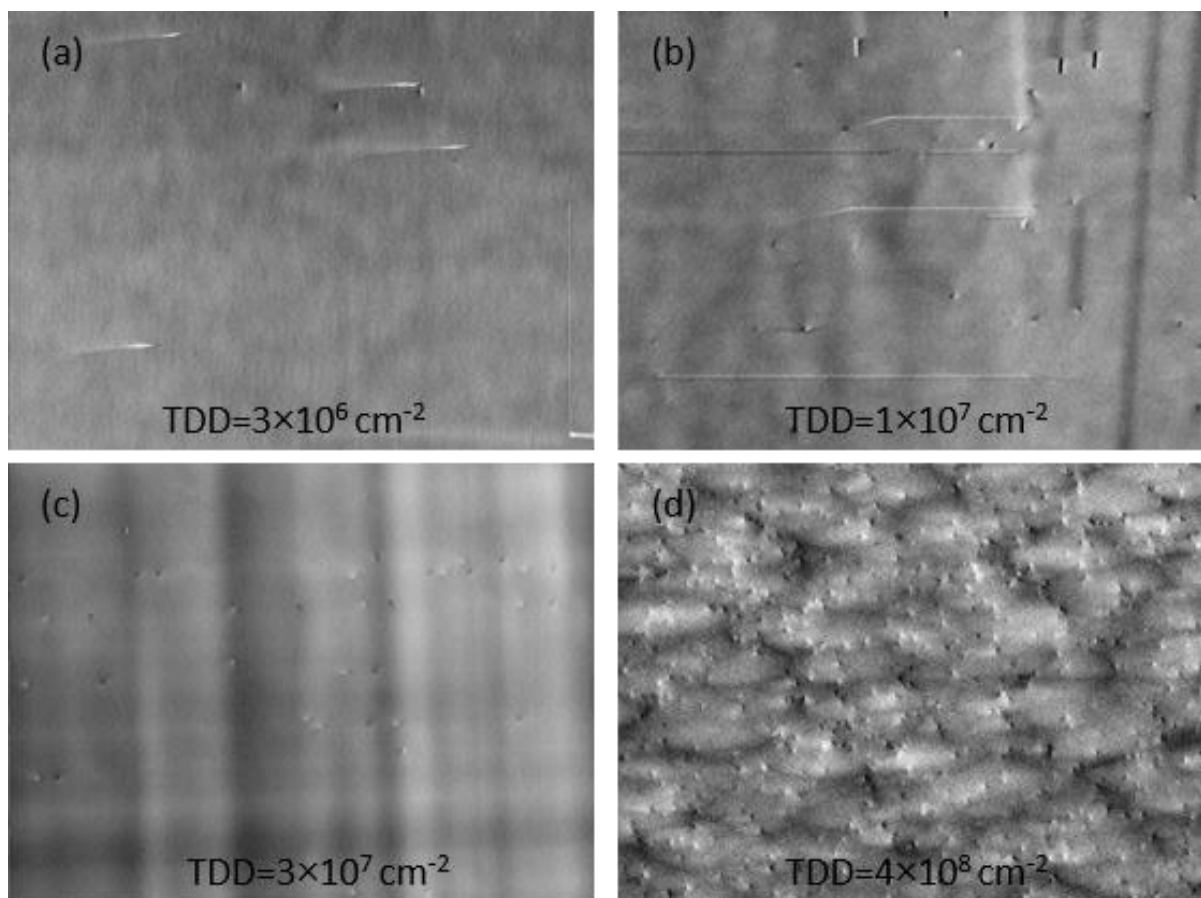


Figure 33: Usability of ECCL over two and a half orders of magnitude of threading dislocation densities from 3×10^6 to $4 \times 10^8 \text{ cm}^{-2}$.

When the TDD is very low it is hard to tell if you are in the right condition. Maybe the conditions are perfectly fine but there is just no dislocation present in the area the experimenter is looking at.

In Figure 34 absolute values for the TDD obtained from different analytical techniques are compared with each other. The red dots represent the TDD obtained from etch pit density investigations, the olive dots represent results from planar view TEM measurements and the black squares represent the measured TDD values from ECCI. The values obtained from ECCI measurements tend to be higher than others. Probably because of some surface defects that are also included in the total number of dislocations even when the features are no dislocations. As mentioned before, the total TDD obtained from EPD might be a little too small, because agglomerations of dislocations are hard to analyze. When the etch pits become too big, a number of dislocations agglomerated in a small area might look like one dislocation/one etch pit. Using PVTEM it is very time consuming to analyze tens of images of small areas to get statistically significant values for the TDD. In all these methods it is hard to get statistically significant average values for the TDD for the whole sample. From the author's experience, experimenters tend to take images of areas with a lot of or very few dislocations. It seems to be some biasing there to search for the right/expected areas.

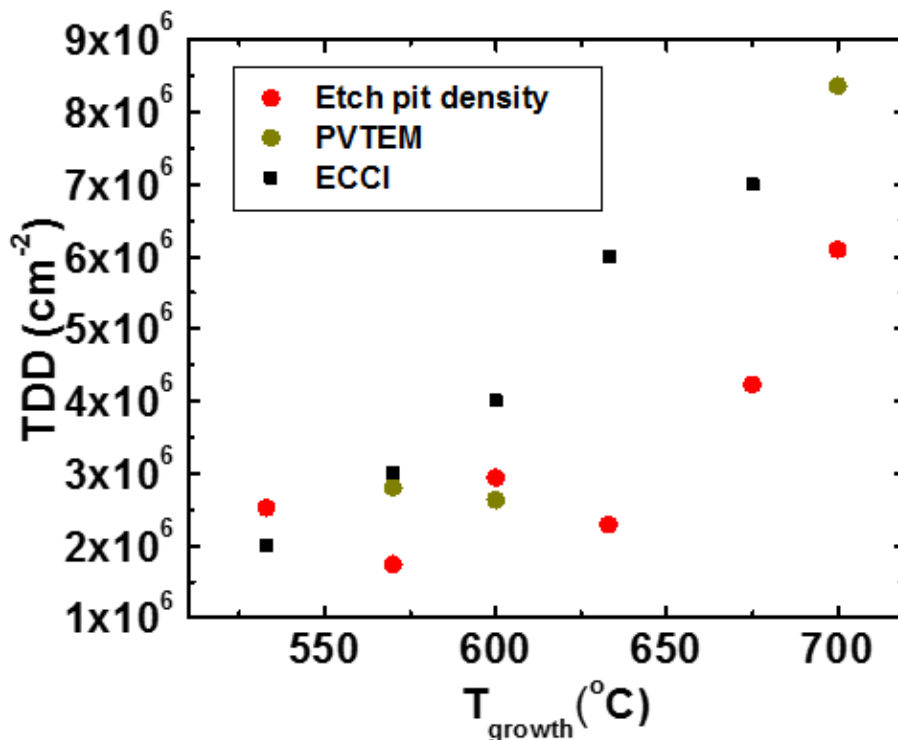


Figure 34: Resulting TDD from EPD (red), PVTEM (olive) and ECCI (black) investigations.

GaAs-Solar Cells on GaP and GaP/Si

Various solar cell structures have been investigated during this project using ECI or EBIC measurements. The threading dislocation density is a dramatically important parameter for solar cells, because every additional recombination center in the form of a defect steals away charge carriers that would otherwise get collected by the electrodes and would increase the efficiency of the solar cell. Very often, the dislocations in a cell are not homogeneously distributed and therefore it is hard to include an efficiency reducing factor for them. Actually, there are controversial discussions in the community how homogeneous or non-homogeneous threading dislocation distributions can affect the device performance differently. In the two following images of GaAs solar cells grown on GaP and GaP/Si substrates the threading dislocations are homogeneously distributed and the TDD was found to be a little lower in the cell grown on GaP/Si. In Figure 35 the top layer of the GaAs grown on GaP cell is shown. Here 164 threading dislocations have been counted. This results in a TDD of $5.38 \times 10^8 \text{cm}^{-2}$.

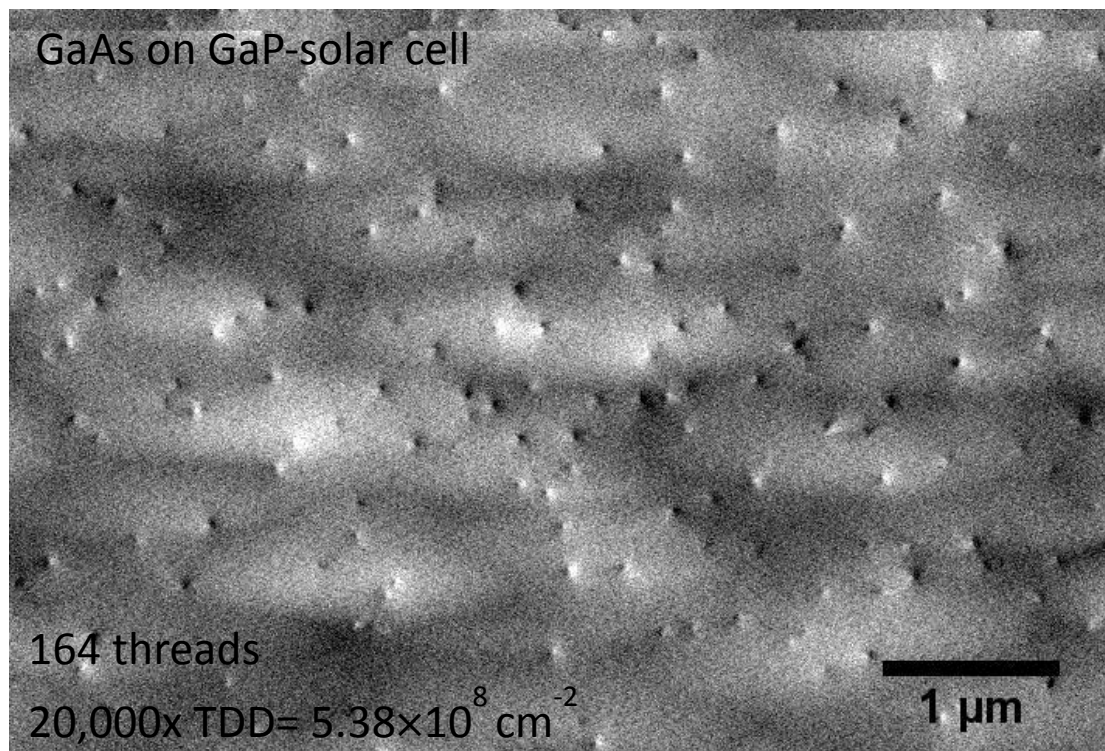


Figure 35: Solar cell structure of GaAs grown on GaP.

Keeping the growth conditions constant (rates and temperatures), in Figure 36 the GaAs top layer solar cell grown on GaP/Si substrate is shown. Here 151 threading dislocations have been counted, resulting in a TDD of $4.95 \times 10^8 \text{ cm}^{-2}$. This shows a slight improvement in comparison to the cell grown on GaP. Nevertheless, TDDs in this range are way too high for solar cell applications. Whether dramatic improvements can be archived by changing the growth conditions or other substrate/top layer material have to be used.

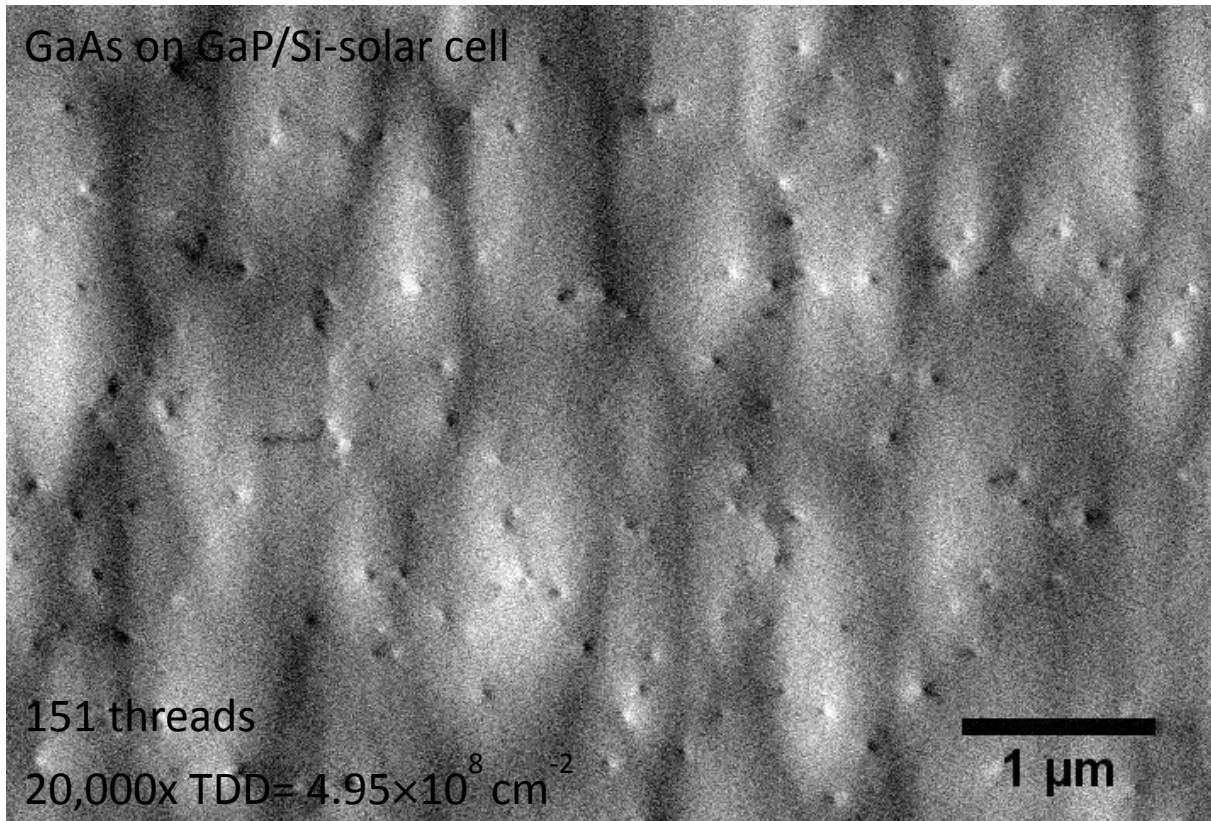


Figure 36: Solar cell structure of GaAs grown on a GaP/Si substrate layer.

Chapter III: Summary, Discussion and Outlook

In this short summary, discussion and outlook the most important achievements of this six months research stay should be reviewed. The importance and output of the work will be analyzed and some future interesting studies will be introduced.

Summary and Discussion

Building up the setup and the evaluation of the analytical technique Electron Channeling Contrast Imaging helped the group at Yale University a lot to improve their theoretical and physical understanding of the channeling process and to value the input resulting from ECCI investigations.

Like every analytical experimental technique ECCI is not perfect and it is important to understand the technique in detail before the channeling experiments can be used as a standard tool. The adjustment process, for a so called controlled ECCI measurement c-ECCI is way more difficult than people would expect from how it is explained in recent papers in literature. Especially when no selected area channeling pattern (SACP) nor beam rocking is possible with the scanning electron microscope (it is actually rather uncommon to have these features in a SEM) adjusting the electron beam exactly to the edge of the chosen Kikuchi line can be a challenge. It demands a lot of experience and skills from the experimenter to adjust it properly to get the right channeling condition. This enables it to compare experimental measurement results with each other and with results obtained with other analytical techniques. In a lot of the samples, that were investigated, big variations in the measured defect density were observed, when the same sample was adjusted in a high symmetry (e.g. (220)) or low symmetry (e.g. (260)) channeling condition. To stay consistent most of the measurements, where it is not stated otherwise, have been performed with the e-beam adjusted to the edge of the (220) Kikuchi line. Some future studies would be interesting to analyze the burgers vector dependency of the threading dislocations in the different investigated materials.

The investigations of various III/V semiconductor materials and device structures showed that an optimized ECCI setup measuring channeling contrast in the (220) channeling condition delivers comparable results for the threading dislocation density like using EBIC, TEM or EPD. In comparison to these techniques it seems that the absolute value of defects measured with ECCI is higher. Until now, we think this might be because of the high surface sensitivity of the method and some surface defects are added to the number of dislocation. But after all, when

used the right way, ECCI seems to be a very powerful method for semiconductor investigations. TDD investigations can be performed over 2-3 orders of magnitudes. There is no built in electric field (e.g. a *pn*-junction or a Schottky contact) necessary. The method is non-destructive and can be performed very fast. For the measurement “only” a SEM is necessary, and no sample preparation is necessary what makes the whole investigation pretty cost effective. Especially for groups that are focused on growth: ECCI measurements are non-destructive and can be performed after every growth step. This can be useful to point out at which step threading dislocations are nucleated.

Future Studies

In the future, ECCI studies obtain with ECP and SACP should be compared in detail. The controlled mechanism to adjust the electron beam to the edge of a Kikuchi line is very important to obtain reliable channeling contrast, so the superior SACP method to adjust the beam should be compared to alternative methods. Experiments like this have been done in literature, but the focus should be concentrated on absolute concentrations or in the comparison of one specific area to be more exact. Otherwise it will be hard to make strong conclusions.

During the experiments presented in this study, a FEG-SEM in an environmental – SEM was used. This sort of setup is normally used to investigate biological or geological specimens and is therefore not optimized for high resolution imaging. Also the backscatter detector was a 2-diode pole-piece mounted detector. A FEG-SEM with a higher special resolution and a better backscatter detector (4-diode detector of the newest generation or a solid state detector) should be compared to the recent results. It might just improve the image quality, but maybe also some surface defects could be identified easier and are not added to the total number of threading dislocations.

Other defects, but dislocations and anti-phase domains should be investigated. The defects have to have some symmetry in the crystal lattice and have to be bigger than so called point defect complexes to obtain channeling contrast. It is not possible to visualize point defects or extended voids with ECCI, because of the limited resolution of a FEG-SEM in comparison to a TEM. Some defects that form due to implantation or relaxation in the lattice could be interesting. Due to heavy proton implantation into silicon, plate like defects structures are formed due to implantation damage and lattice stress relaxation. According to literature,

these defects are oriented in special crystal directions e.g. (111), (110) so it should be possible to investigate these defect complexes.

The surface preparation seems to be a very important factor to perform reliable ECCI measurements. For a lot of as grown structures/devices it is no problem to investigate the top layers without extra sample preparation. But, especially for novel device concepts and new growth plans there can be rough top layers with a lot of defects in it. The quality of the top layer is normally checked with AFM. Several efforts have been performed by grinding and polishing to improve the surface quality. Also ion milling/sputtering off a very thin surface layer was performed by Martin Faccinelli. According to Prof. S. Zaefferer this problem is already controlled pretty well in their group for metals. Future surface preparation studies should enable the investigation of very rough samples too.

It would also be very interesting to perform ECCI on the cross section of a device. It was not possible to obtain a good ECP image and adjust the electron beam to a Kikuchi line in the authors setup, but maybe with a SACP setup this could be possible and would tell the experimenter more about the defect properties of the different layers of a device.

Bibliography

- Trager-Cowan, C. et al. (2007). Electron backscatter diffraction and electron channeling contrast imaging of tilt and dislocations in nitride thin films. *Physical Review B*, p. 75(8).
- Wilkinson, A. J. et al. (1993). Electron channeling contrast imaging of interfacial defects in strained silicon-germanium layers on silicon. *Philosophical Magazine A*, pp. 68(1) 59-80.
- Bieler, T. R. et al. (2009). The role of heterogeneous deformation on damage nucleation at grain boundaries in single phase metals. *International Journal of Plasticity*, pp. 25.9 1655-1683.
- Booker, G. R. et al. (1967). Some comments on interpretation of Kikuchi-like reflection patterns observed by scanning electron microscopy. *Philosophical Magazine*, pp. 16(144) 1185-1191.
- Carnevale, S. D. (2014). Rapid misfit dislocation characterization in heteroepitaxial III-V/Si thin films by electron channeling contrast imaging. *Applied Physical Letters*, pp. 104, 232111.
- Clarke, D. (1971). Observation of crystal defects using scanning electron microscope. *Philosophical Magazine*, pp. 24(190) 973-979.
- Coates, D. G. (1967). Kikuchi-like reflection patterns obtained with scanning electron microscope. *Philosophical Magazine* 16(144), pp. 1179-1185.
- Czernuszka J. T. et al. (1990). Imaging of dislocations using backscattered electrons in a scanning electron-microscope. *Philosophical Magazine Letters*, pp. 62(4) 227-232.
- Gutierrez-Urrutia, I. S. et al. (2010). The effect of grain size and grain orientation on deformation twinning in a Fe–22wt.% Mn–0.6 wt.% C TWIP steel. *Materials Science and Engineering: A*, pp. 527.15 3552-3560.
- Picard, Y. N. et al. (2007). Electron channeling contrast imaging of atomic steps and threading dislocations in 4H-SiC. *Applied physics letters*, p. 90.23 234101.
- Picard, Y. N. et al. (2007). Nondestructive analysis of threading dislocations in GaN by electron channeling contrast imaging. *Applied Physics Letters*, p. 91.9 094106.
- Simkin, B. A. et al. (2003). A factor to predict microcrack nucleation at γ - γ grain boundaries in TiAl. *Scripta materialia*, pp. 49.2 149-154.
- Simkin, B. A., & Crimp, M. A. (1999). An experimentally convenient configuration for electron channeling contrast imaging. *Ultramicroscopy*, pp. 77(1-2) 65-75.
- Stern, R. M. et al. (1972). Dislocation images in high-resolution scanning electron-microscope. *Philosophical Magazine*, pp. 26(6) 1495-1499.