Załącznik 4

## Sławomir Kret

## Institute of Physics Polish Academy of Science

# Summary of Professional Accomplishments

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## 1. Education and Scientific degrees

1998 doctoral	Université Pierre et Marie Curie PARIS 6, Paris, in Solid State Physics, France, PhD thesis on: <i>Analyse quantitative à l'échelle atomique par</i> <i>traitement d'images de microscopie électronique "haute résolution" dans</i> <i>des hétérostructures III-V et II-VI fortement désadaptées</i> <sup>1</sup> Supervisors: Dr Clode Delamarre and Prof. dr hab. Jacek Kossut In 1999 the degree was recognized as an equivalent of a degree of doctor of physical sciences by the Scientific Council of the Institute of Physics Polish Academy of Sciences.
1989 higher	Warsaw University of Technology, Faculty of Materials Engineering Master's thesis: "The structure of multi-compound metallic coatings obtained by the pulse plasma method based on the example of selected high- temperature alloys", Supervisor: Prof. dr hab. Krzysztof Zdunek
1983 secondary	

## 2. Information on previous employment in scientific institutions

since 1998-present	Institute of Physics PAS in the Electron Microscopy Group as: physicist,
	assistant, assistant professor
1993-1998	Parallel doctoral studies at the Institute of Physics PAS and Université
	Pierre et Marie Curie PARIS 6
1989-1992	Scientific-Production Center of Semiconductors in Warsaw, Department of
	Design and Technology of Discrete Components as Lead Technologist

<sup>&</sup>lt;sup>1</sup> English translation: *Quantitative analysis of stress relaxations at the atomic scale by image processing of HRTEM images in highly strained quantum III-V and II-VII heterostructures* 

## 3. Information on published research papers and creative professional work

Scientific output includes 93 original works, including 87 after completion of the PhD According to the ISI Web of Science database (14.05.2012)

Hirsch Index: H=12

Total number of publications: 86

Total number of citations: 553 (456 without auto citations)

Current version of the Web Of Science statistics: <u>http://www.researcherid.com/rid/D-5585-2012</u> Total impact factor: **122.1** 

The works have been published in referred physical journals and specialized journals of high international reputation in nanotechnology, microanalysis and electron microscopy:

1. Applied Physics Letters: 5

- 2. Journal of Applied Physics: 5
- 3. Journal of Physics : Condensed Matter: 2
- 4. Ultramicroscopy: 1
- 5. Microscopy and Microanalysis: 2
- 6. Japanese Journal of Applied Physics: 1
- 7. Philosophical Magazine: 2
- 8. Nanotechnology: 5
- 9. Nano Letters: 2
- 10. Micron: 1
- 11. Journal of Microscopy Oxford: 2
- 12. Physica Status Solidi A, B: 6

The full list of publications and conference presentations in English is in Annex 6.

A total of 134 conference presentations (120 after completion of the PhD):

- a) 11 invited lectures, 6 delivered in person after PhD completion
- b) 62 oral presentations, 55 after PhD completion, 17 delivered in person
- c) 71 poster presentations, 64 after PhD completion
- d) Total number of delivered seminars: 16

## 4. Indication of the main scientific achievement

The main scientific achievement according to the provisions (as stipulated by Act. the Act: Dz. U. nr 65, poz595 as of march 14, 2003 with changes) consist of the publication list under title:

# Quantitative high resolution transmission electron microscopy in physics and technology of semiconductor heterostructures.

- H1 "Measurement of dislocation core distribution by digital processing of high-resolution transmission electron microscopy micrographs: a new technique for studying defects", <u>S. Kret</u>, Paweł Dłużewski, Piotr Dłużewski, E. Sobczak, Journal of Physics: Condensed Matter, vol. 12, no. 49, pp. 10313-10318, 2000
- H2 "On the measurement of dislocation core distributions in a GaAs/ZnTe/CdTe heterostructure by high-resolution transmission electron microscopy", <u>S. Kret</u>, Paweł Dłużewski, Piotr Dłużewski, J-Y. Laval, Philosophical Magazine, vol. 83, no. 2, pp. 231-244, 2003
- H3 "Analysis of strain in the {1120} prismatic fault in GaN using digital processing of high-resolution transmission electron microscopy images", <u>S. Kret</u>, P. Ruterana, G. Nouet, Journal of Physics: Condensed Matter, vol. 12, no. 49, pp. 10249-10256, 2000
- H4 "Investigation of threading dislocation atomic configurations in GaN by HRTEM, geometrical phase analysis and atomistic modelling", <u>S. Kret</u>, J. Chen, P. Ruterana, G. Nouet, Institute of Physics: Conference Series, vol. 169, pp. 319-322, 2001
- H5 "The dislocations of low-angle grain boundaries in GaN epilayers: a HRTEM quantitative study and finite element stress state calculation", <u>S. Kret</u>, P. Dłużewski, G. Maciejewski, V. Potin, J. Chen, P. Ruterana, G. Nouet, Diamond and Related Materials, vol. 11, pp. 910-913, 2002
- H6 "Piezoelectric field around threading dislocation in GaN determined on the basis of high-resolution transmission electron microscopy image", G. Maciejewski, <u>S. Kret</u>, P. Ruterana, Journal of Microscopy Oxford, vol. 223, no. 3, pp. 212-215, 2006
- H7 "Quantitative measurement of In fluctuation inside MOCVD InGaN QWs", P. Ruterana,
  <u>S. Kret</u>, M.A. Poisson, Materials Science and Engineering B, vol. 93, no. 1-3, pp. 185-188, 2002
- H8 "Composition fluctuation in InGaN quantum wells made from molecular beam or metalorganic vapor phase epitaxial layers", P. Ruterana, <u>S. Kret</u>, A. Vivet, G. Maciejewski, P. Dłużewski, Journal of Applied Physics, vol. 91, no. 11, pp. 8979-8985, 2002,

- H9 "Modelling of indium rich clusters in MOCVD In<sub>x</sub>Ga<sub>1-x</sub>N/GaN multilayers", G. Jurczak,
  G. Maciejewski, <u>S. Kret</u>, P. Dłużewski, P. Ruterana, Journal of Alloys and Compounds, vol. 382, pp. 10-16, 2004
- H10 "Contribution to quantitative measurement of the In composition in GaN/InGaN multilayers", <u>S. Kret</u>, G. Maciejewski, P. Dłużewski, P. Ruterana, N. Grandjean, B. Damilano, Materials Chemistry and Physics, vol. 81, pp. 273-276, 2003
- H11 "Homogenous indium distribution in InGaN/GaN laser active structure grown by LP-MOCVD on bulk GaN crystal revealed by transmission electron microscopy and x-ray diffraction", <u>S. Kret</u>, P. Dłużewski, A. Szczepańska, M. Żak, R. Czernecki, M. Kryśko, M. Leszczyński, G. Maciejewski, Nanotechnology, vol. 18, pp. 465707 (9), 2007
- H12 "Inhomogeneities of InGaN/GaN MOVPE multi quantum wells grown with a two temperatures process studied by transmission electron microscopy", <u>S. Kret</u>, F. Ivaldi, K. Sobczak, R. Czernecki, M. Leszczyński, Physica Status Solidi A, vol. 207, no. 5, pp. 1101-1104, 2010
- H13 "TEM characterization of MBE grown CdTe/ZnTe axial nanowires", P. Dłużewski,
  E. Janik, <u>S. Kret</u>, W. Zaleszczyk; D. Tang, G. Karczewski, T. Wojtowicz, Journal of
  Microscopy, vol. 237, no. 3, pp. 337-340, 2010

Physics and technology of semiconductor devices have mutually stimulated each other's development. Presently, individual elements of the active structures reach nanoscale dimensions, including application of materials with different ban gaps and physical properties. Achieving a high level of performance for commercial devices requires an understanding of the subtle phenomena occurring on the nano and atomic scales, which can change quantum effects used in generating and detecting light or influencing the process of carriers transport in transistor gates. The key issue is the quantitative description of local structures and understanding their influence on properties in this scale. A High Resolution Transmission Electron Microscopy (HRTEM) can directly deliver such information at the atomic level. My scientific activity concentrated on the development of methods for analyzing and interpreting HRTEM, permitting delivering the reliable quantitative data and application of such methodology in physics and technology of semiconductor heterostructers. This work is part of a more general scientific study:

- 1. Development of microscopic methods of chemical element analysis and local structure at the atomic level suitable for quantum nanostructures.
- 2. Understanding the accommodation process of lattice mismatch in buffer layers and minimization of the defect density in unmatched heterostructures.
- 3. Understanding the relationship between the atomic structure of dislocation cores and their efficiency in non-radiative recombination of carriers.
- 4. Identification of the structural origins determining the efficacy of lasers and LEDs based on nitride semiconductors.

- Improvement of efficiency and enlargement of the spectral range of light emitters by modifying the structure of the interfaces and spatial distribution of the indum atoms inside InGaN/GaN QWs
- 6. Development of a new generation of sensors of environmental conditions and photon light emitters based on axial and core shell nano-wires ZnCd(Mg,Mn)Te.

Presently, physics and technology of semiconductor nanostructures based on III-V semiconductor compounds (GaAs,InGaAs), nitride semiconductors (GaN, AIN, InN), or II-VI ((CdTe, ZnTe(Mn, Mg), employ different epitaxial techniques. The most important technologies are based on Molecular Beam Epitaxy (MBE) and Metalo Organic Vapour Phase Epitaxy (MOVPE) or their different variants and derivatives. Heterostructures based on ternary alloys of semiconductor compounds bring about the possibility of attaining most of the desired properties. Unfortunately, some physicochemical characteristic of materials considerably limit freedom in mixing and linking different materials. Materials with different band gaps may have different lattice parameters, partial pressure, coefficients of thermal expansion, and elastic constants. Therefore, the fabrication of heterostructures will be associated with the appearance of structural defects as well as imperfections of interfaces. From the thermodynamical point of view, most of the semiconductor alloys are unstable and have a tendency to phase separation, as for example spinodal decomposition in the case InGaAs, InGaN or InAIN.

In addition, epitaxial growth is a highly non-equilibrium process. The structure of the growing layer depends on thermodynamic and kinetic conditions. Moreover, surface processes, which take place on the solid vapor/vacuum interface, can be very important. Finally, the resulting structure may deviate significantly from an ideal monocrystal. The heterostructure properties are highly modified by defects like dislocations, stacking faults, inversion domains, twining, low and high angle grain boundaries of mosaic islands, non-equilibrium point defects, internal stress fields and linked electrical fields related to the piezoelectric nature of materials. Consequently, carrier mobility may drop, since defects act as non-radiative recombination centers.

In the technology of epitaxial growth, basic characterizations are performed with the use of atomic force microscopy (AFM), X-ray diffractometry and reflectometry as well as optical methods such as PL and Raman Spectroscopy. AFM gives information about surface structures, X-ray and optical methods deliver precise information about chemistry and interfaces but averaged based on large volumes of material. A Transmission Electron Microscopy (TEM) gives access to the local structure inside the material within very small volumes. The target of quantitative TEM methods are not only visualization of selected structural features but also numbers that can be confronted with theoretical models and macroscopic measurements.

## Main achievements of the PhD research work:

- 1. Elaborating on the methods of automatic detection and quantitative analysis of extended configurations of the defects based on the dislocation density tensor (H1, H2).
- 2. Proving that accommodation of the lattice mismatch mainly occurs at the CdTe/GaAs interface through the network of Lomer dislocations having dissociated cores (H1, H2).
- 3. Proving that the translation vector of the prismatic stacking error in the GaN may change under stress, and that errors may occur on the level of location and cause dislocation with blurred cores (H3).
- 4. Practical realization of the concept of transferring the experimental configuration of the defect onto the Finite Element mesh in order to analyze its interaction (H5, H6).
- 5. Proving that most experimentally observed dislocation core structures on highly defected crystals buffer layers differ significantly in structure from theoretical models of equilibrium atomic configuration (H1-H6).
- 6. Proving that the potential of edge threading dislocation in GaN caused by the piezoelectric effect related to distortion field around a core is small in relation to the total potential reported from holographic experiments. This means that the origin of this potential might be broken bounds or doping of the core (H6).
- 7. Developing methods of determining the absolute indium concentration quantity based on the lattice distortion on HRTEM images and modeling of the relaxation on the thin cross section with the use of experimentally determined geometric parameters (H7, H8, H10, H11).
- 8. Development of methods to determine the ultrathin TEM foil thickness (lower than 30 nm) for GaN electrons in double beam conditions, based on the thickness fringes for non axial illumination (H11).
- 9. Determining, on the basis of the simulation with the use of atomic models of the heterostructures and the dynamical theory of electron scattering, optimal conditions for performing high resolution electron microscopy observations that minimize the delocalization effects and modification of displacement fields for CdTe/GaAs and GaN/InGaN heterostructures (H2, H4, H8, H9, H10, H11).
- 10. Detection of the differences in concentration and distribution of indium atoms between the InGaN QWs in the frame of a single active region-MQW(multi quantum wells) (H7, H8, H11).
- 11. Proving that during the MOVPE process, InGaN QWs grew with nominal indium concentrations at a 15% level, and that indium rich burned clusters measuring 1-3 nm can be created (H7,H8,H9).
- 12. Demonstrating that during the dual temperature MOVPE process, desorption of indium occurs with uncovered QW, leading to surface steps and plane stripes forming having an anisotropic morphology (H12).
- Determining the width of the transition region in axial catalytic ZnTe/CdTe nanowires (H13).

## 4.1 A guide to the monothematic cycle of papers.

## 4.1.1 Initial State of Knowledge

Currently, information about chemistry with nanometric or sub-nanometric spatial resolution can be obtained by different TEM techniques as EDS spectroscopy, EELS, and Z-contrast. Aberration correctors are now popular. However, access to these methods as well as their resolution was limited at the end of the 20<sup>th</sup> century. Spectroscopic methods hardly achieved a resolution of a few nanometers. Moreover, determining the distortion field on the basis of diffraction contrast was limited by the difficulties and errors in absolute contrast measurement, and spatial resolution was limited to a few nanometers.

At the same time, high-resolution transmission electron microscopy (HRTEM), which uses phase contrast, achieved atomic resolution in a few high voltage microscopes. However, highly energetic beams of 1-3MeV caused heavy radiation damage of semiconductor materials. Accessible in many research centers, microscopes with acceleration voltage in the range of 200-300kV achieved point resolution in the range of 0.2 nm, making possible to obtain "structural images" of many semiconductors (expect diamond). In the case of semiconductors with sphalerite structure, it was possible to obtain images of the 111, 002 and even 220 lattice fringes , and in the case of wurtzite structure images of 0002, 10-10 lattice fringes. Such images contain information about local lattice distances.

During this period, the measurement of the local lattice period was applied for the first time to evaluate the chemical composition of quantum wells using a computer analysis of HRTEM images and application of Vegard's law [1] for ternary semiconductors. In addition, my scientific activity during the preparation of the PhD was related to the methodology of such measurements performed on individual micrographs (see 5.1.3). Because microscope investigation is performed using a thin cross-section of the heterostructure after partial relaxation occurred during the thinning process, it is necessary to take this effect into account. Together with the group of prof. A. Lefebvre from Lille University, we started the analysis of thin foil relaxation using finite element modeling. Related work was published after my PhD defense [2]. A good summary of the state of the art at the beginning of 21st century is given in the review paper by Kret et al. [3].

The precision of such kinds of distortion measurement depends on the conditions of the HRTEM experiment. This is particularly true in the case of **noncentrosymetric crystals** such as non-random ternary semiconductors (in the case of SiGe alloy this problem is less critical). The problem of influence of the gradient of microscope contrast transfer function was announced for the first time at a conference paper [4],in which I'm coauthor. Imperfections and aberrations of electron optics are some of the most important sources of errors causing delocalization of strain information on the HRTEM images. Averaging the information across the whole TEM thin foil and loss of information along the electron beam is the next problem we need to solve. The

aspects related with understanding of such a phenomena and their impact on quantitative measurements was the object of my scientific work for such measurements because they strongly affect the reliability of results.

## 4.1.2 Defects and their Spatial Configurations in Buffer Layers

For epitaxy of many semiconductor materials, cheap and sufficiently large lattice matched substrates are missing. For this reason, significantly great research effort is devoted to developing heteroepitaxial growth on foreign substrates, which have different lattice constants and occasionally different crystal structures. Successful growth of the thick layers and quantum structures with relatively low defect densities have already been demonstrated. One of the examples is the growth of heterostructures based on the CdTe/ZnTe system on GaAs substrate by Molecular Beam Epitaxy (MBE). Lattice mismatch between CdTe and GaAs is at around the level of 15%.

One of the strategies of defect reduction is growing the buffer layer sufficiently thick to permit the defects to react and annihilate. The best solution is the accommodation of lattice mismatch between layer and substrate directly directly on interface by a network of misfit dislocations. Such dislocations cannot propagate into the buffer layers. The efficacy of such lattice mismatch accommodation depends of the types of dislocations.

In the case of sphalerite structures, the most effective accommodation is achieved by  $90^{\circ}$ Lomer type dislocations having well determined atomic structures of the core 5/7 [5]. The other  $90^{\circ}$  dislocation with irregular core structure and the pairs of  $60^{\circ}$  dislocations are less effective. Isolated  $60^{\circ}$  dislocations are undesired due to the fact that they can propagate to the layer and cause local lattice rotation.

The formation mechanism of a given type of misfit dislocations is still an open scientific and technological question. It is possible to drive of the topology of the misfit dislocation networks well as structure of the cores by applying different kind of "technological trick". Substrates with different types of atomic steps, doping, annealing, etc. are applied. HRTEM microscopy permits analyzing the misfit dislocation network on the cross-section of interfaces between the substrate and epilayer and gives us access to the core structure.

The problem is that the HRTEM image cannot be simply interpreted as a projection of the crystal structure. Indeed, such images from the phase contrast are intensity maps of the interference field from the electron wave passing through the crystal and modified by the optical system of the microscope[6].

Particularly complicated is interpreting the image of non-periodic objects, such as dislocations and phase boundaries. One of the approaches for solving this issue is through computer simulations, amplitude and phase reconstruction from a series of images obtained from different focuses of objective lenses or tilts of incident electron beams. However, all these approaches are rather complicated and difficult to apply to systematic investigations related to optimization of epitaxy processes.

In the [H1] paper, the concept that the **Burgers vector of dislocations as well as that the core structure can be determined through the analysis of the symmetry of lattice distortion generated in the close proximity of the dislocation core was proposed.** Measurement of the lattice distortion on the HRTEM images can be performed by different methods [3]. Up to date, the Geometric Phase Analysis (GPA) proposed by M.Hytch et al. [7] is the most useful method suitable for images with defects.

The method is adopted for nanoscale analysis, being a computer-based version of the Moire method applied in mechanics for deformation measurement starting since 1948 [8]. In this method, precision shifts of the lattice fringes of deformed crystals are measured in relation to perfect reference lattice fringes. In practice, this method is performed in Fourier space (i.e. diffraction spots or Bragg reflexes corresponding to lattice fringes).

The measurement of displacements for two different crystallographic direction permits to determine a two dimensional tensor of lattice distortion. In my work [H1], this method was applied for the first time for measurement of the distortion field at areas with a network of misfit dislocations. The reconstruction of the lattice distortion tensor  $\beta$  was based on two sets of 111 lattice fringes.

For analysis, selected region of CdTe/GaAs interface have homogenous thickness on both sides of boundary. In the case of the crystal thickness of about 10 to 15 nm and Scherzer defocus of objective lens the maximum of the contrast transfer function of this microscope is near to spatial frequencies corresponding to 111 interplanar distances. This condition gives the best possible S/N ratio and the phase shift due to gradient of CFT for these spatial frequencies are minimized.

Finally, four components of the lattice distortion tensor  $\beta$  are obtained. The distortion fields around each dislocation differ significantly. At the same time, Burgers vectors determined by the Burgers contour show that all dislocations are Lomer dislocations with the Burgers Vector of a/2[1-10]. In discussions with Professor Paweł Dłużewski, who is a specialist of the continuum theory of dislocations, we concluded that on the basis of the tensor of lattice distortion  $\beta_{ij}$  it was possible to calculate the dislocation density tensor  $\alpha$ . This tensor was

introduced nearly 50 years ago by Nye [9] and defined as:  $\tilde{\alpha} = -curl\beta$ 

Based on the visualizations of the dislocation density, tensors  $\alpha_{13}$  and  $\alpha_{23}$  are represented as small peaks with diameters of about 0.2 nm. Components of this tensor are calculated in a coordinate system related to pixels of microscopic images. Because the Burgers vector in continuous dislocation theory is defined as a surface integral in core region Sc:  $\hat{b} = -\int_{Sc} \tilde{\alpha} ds$  Then the in plane component of Burgers vector can be calculated as a sum of all pixels in the core region. Independently of possible differences in the distributions and the shapes of maxima/minima of dislocation density tensor calculated in such way, the Burgers vector components are identical for each dislocation and corresponds to Burgers vectors of the Lomer dislocation.

At the interface of the GaAs/CdTe(Zn), we have an abrupt change of chemistry, but the continuity of the crystal lattice is conserved, therefore, we need to describe this situation using a **"chemical distortion tensor."** For this reason, in paper [H2], starting from the basis of theory of large deformations, the term "true Burgers vector "was introduced to take into account such changes in the chemistry of heterostructures and made it possible to calculate the module of the vectors related to the net reference structure<sup>†</sup>.

The distribution of dislocation density tensors in the core regions presented for proximal dislocations shown in work [H2] are significantly different. After integration, identical values of the true Burgers vectors are obtained for each dislocation. Only one dislocation D11 along the analyzed 50 nm long interface region is a 60° dislocation (another 60° dislocation is located on the right side above the interface). Also, dislocation D5 has compact cores – perfect Lomer type dislocations. On the other hand, dislocation D7 can be interpreted rather as a pair of two 60° dislocations. As shown, even when the resolution of the microscope is insufficient for resolving the atomic structures of the cores, it is possible to distinguish between different core structures on the basis of the dislocation density tensor distribution.

Development of the methods of determining the dislocation density tensor on the basis of the HRTEM image was performed in the framework of two KBN Projects [Pro2, Pro3]. The results of my measurements of lattice distortion for the epitaxial system GaAs/ZnTe/CdTe were used in subsequent works [10,11] devoted to calculating the stress fields for this dislocation network. In the range of high deformation, the Hook law is invalid: most materials have greater stiffness for compression than for dilatation. Due to the application of nonlinear mechanic approaches and utilization of the high order stiffness coefficient, it was possible to correctly calculate stress distribution. The experimental values of distortion tensor  $\beta$  were attributed to the nodes of the finite element mesh. As a result of iteration, we obtained the stress field distribution for the real configuration of the dislocation network, which showed that such dislocation introduced a global expansion at this heterointerface. This nonlinear effect is important from the practical point of view when the macroscopic wafer bending is analyzed. This methodology of dislocation density tensor measurement proposed in works H1 and H2 can be used as a generator of realistic configurations of dislocation suitable in modeling heteroepitaxial growth.

The second investigated strained heteroepitaxial system were the GaN layers on SiC and Al<sub>2</sub>O<sub>3</sub> substrates. SiC substrates are particularly well located in high power devices for

<sup>&</sup>lt;sup> $\dagger$ </sup> For example, the b<sub>1</sub> component for dislocation D1 in actual configuration has a value of -0.454 nm, which becomes -0.401nm in reference to GaAs, where this corresponds exactly to the Burgers vector of such a dislocation in the GaAs net. On the other hand, if we compare -0.454 nm with the Burgers vector of the dislocation in CdTe (-0.4583 nm), then we are led to conclude that we have a CdZnTe alloy core near the dislocation. The Zn atoms there form a very thin (a few monolayers) nucleation layer of ZnTe, which has a smaller lattice mismatch as compared to GaAs.

applications in high temperature environments. The lattice mismatch between GaN and 6H-SiC is in the order of 3.4%. In the case of sapphire substrates, the mismatch is higher by  $\sim$ 16%, but the advantage is the relatively low price and accessibility of such substrates.

An analysis of the structural defects in the GaN and AlN epilayers was the subject of scientific works of the Directeur de la Recherche (DR) Gerard Nouet group started in 1995 in the CRISMAT-LERMAT laboratory in Caen in France. My contribution consisted mainly of introducing into defect analysis quantitative methods for analyzing HRTEM images during my post doctorate stay in this laboratory in the framework of the European RTN program IPAM (HPRN-CT-1999-00040 (*IPAM Interface Analysis at Atomic Level and Properties of Advanced Materials*) coordinated by Directeur de Reserch (DR) Pierr Ruteran.

Presently, most of the authors, mainly on the basis of computer modeling, but also based on experimental results, agree that screw and mix dislocations are the more active centers of nonradiative recombination and effectively reduce potential barrier compared to edge dislocations [12, 13]. However, such images are not very coherent. Recent, TEM and cathodoluminescence investigations performed by M. Albrecht et al. [14] suggest that doping can change electrical activity of dislocations, for example, in make edge dislocation of the type  $\vec{a}$  active and mixed  $\vec{a} + \vec{c}$  inactive. Despite this, it is clear that reduction of the dislocation densities improve the properties of devices. The key problem, which is still not solved, is understanding and identifying mechanisms of propagation and annihilation or reaction between defects leading to the reduction of their density.

My work concentrated on analyzing selected configurations of interacting defects in real material. I am not interested in a detailed investigation of the atomic structures of individual dislocation cores **but in catching the structural modifications, which appear when the defect interacts between them**. Local lattice distortion and dislocation density measurements are good utilities permitting the analysis of relatively large areas of defective crystal in a quantitative way.

The work [H3] is devoted to an analysis of the structure of prismatic stacking faults {11-20}, being part of the group of planar defects: translation domain boundaries – TDB. The analysis was performed for two connected and interacting long segments of this planar defect. This PSF starts with a void and is terminated by a partial dislocation. Previous experiments undertaken by the group with Dr. G. Nouet have shown that the atomic structure of the PSF boundary corresponds to Drum's atomic configuration [15, 16] and the total displacement vector of the boundary of Rigid Body Shift RBS is 0.53a [17].

In [H3] work, I have shown that the distortion field appearing during coalescence of the islands can disturb this structure, locally changing the interface shift and leading to the generation of dislocations along the surface of such SF.

In order to be able to carry out the quantitative analysis of HRTEM images, it is necessary to select optimum conditions of observation. In our work [H3], a high-resolution investigation was performed with a Topcon 002B microscope operating at 200kV with a point resolution of 0.18nm. The objective lens of this microscope has a relatively small coefficient of

spherical aberration, 0.4 mm. In HRTEM of GaN in the [0001] zone axis, only  $01\overline{1}0$  type beams and partially  $11\overline{2}0$  beams were used for image formation. An aperture cut out the remaining beams in the back focal plane of the objective lens. Dynamic simulations for perfect GaN crystal as well for defects were performed using multi-slice algorithm and EMS software [18]. The simulations clearly show that in conditions when the bright contrast maxima correspond to the tunnel (low density of electrons), the position of the maxima are stable against variation of the thickness and defocus even in the direct proximity of defect cores.

A local lattice distortion analysis performed for segment SF1 shows that the translation vector modulus of stacking fault is near the theoretical value 0.53a only close to the void where strain relaxation took place. This vector for the remaining part of the SF1 segment changes and reached 0.6a. In the case of the SF2 segment analysis, the dislocation density tensor clearly shows the presence of dislocations with non-localized cores at the fault boundary. This delocalization takes place at a distance of a few lattice constants. The integration of  $\alpha$  fields, which have different shapes and extend the  $\alpha$  peaks, shows that all dislocations D1-D4 have the same in-plane components of Burgers vectors. Based on this analysis of dislocation density tensor, the structural model of the SF2 segment was created.

The example from work [H3] proved that the application on large scale for HRTEM image of the interpretation method based on the dislocation density tensor gives a synthetic diagram even of complex arrays of defects. The results obtained in [H3] prove that real structures deviate significantly from atomic models for such defects obtained through the assumption of a fully relaxed environment. A translation vector larger than the theoretical translation vector of the PSF as well the presence of dislocations in boundaries suggests that the real structure accumulates much more elastic energy that is concluded from models of isolated defects. This work also contributed to the following research supervised by DR Gerard Nouet [19, 20] as well research performed by other groups which tried to estimate of the energy of different TBD in epitaxial layers of GaN [21]. In particular, the calculation of the energy of TBD containing dislocations and localized strain fields is shown for first time by my analysis.

For GaN buffer layers growing along the [0001] direction, the mosaic structure is due to coalescence of the nucleation islands. This process leads to the formation of low and sometimes high angle grain boundaries. Local lattice distortion analysis (LLDA) and dislocation density tensor analysis (DDTA) of HRTEM images in the [0001] zone axis of GaN epilayers after removal of the substrate clearly show the coalescence mechanisms of such islands and arrangements of edge dislocation networks. In works [22] and [H4], I analyzed two islands after coalescence.

The analysis shows that the disorientation of the two different parts of epitaxial layers can change progressively from 2° to 6° at a distance of 20 nm. The increase of disorientation is related to a decrease in the distances between edge dislocations from 2 to 10 nm. V. Potin et al investigated similar but isolated dislocations in their work. [23]. Using one of these core models made of 5/7 atom rings, a supercell containing isolated edge  $\vec{a}$  type dislocation was generated.

This supercell was relaxed with the use the modified Stilinger-Weber empirical potential [24]. I then used the equilibrium atomic positions to calculate simulated HRTEM images using the dynamical theory of electron scattering.

I was then able to compare the components of the distortion field extracted from experimental and simulated images and showed that the values and symmetry of components  $\beta_{11}$  and  $\beta_{22}$  are very similar. In the case of  $\beta_{12}$  and  $\beta_{21}$  compounds (which are related to the rotation of the lattice), the symmetry of the spatial distribution obtained for experimental dislocation from a low angle boundary is highly disturbed in comparison to the model. It is related to the local disorientation of two crystals and interaction with neighboring dislocations.

I analyzed the possibility of determing the core structure on the basis of distribution of the dislocation density tensor in my experimental work [H4] using supercells containing different models of dislocation cores. In this analysis, I took into account the TOPCON 2B microscope with an acceleration voltage of 200KV as well as the high voltage microscope with acceleration voltage of 1MeV. In simulation, I also took the spherical aberration coefficient as a parameter (in this period the first aberration corrected microscope was announced). Experimentally, even for the perfect dislocation cores, two  $\alpha$  peaks were observed. For the 200KeV microscope at Scherzer defocus and for a 10 nm foil thickness, the smallest spreading distance of  $\alpha$  peaks at 0.2*a* was observed for the 7/5 core. For the 4 and 8 atom cores, the determination of the core type using  $\alpha$  peaks is possible only for very limited sets of defocus and thickness of the foil. This is quite easy to do in the case of high voltage microscopes or microscopes with aberration corrected optics.

## The splitting of the $\alpha$ field for theoretical configurations of the dislocation cores are much smaller than those observed experimentally for dislocations on low angle boundaries which are in the range of 1.5-2a (i.e. almost 10x larger). This comparison clearly shows that low angle boundaries are at a high non-equilibrium state: their cores have non-regular structures and probably store large amounts of elastic energy.

The simulation of a system of 14 dislocations with the use of empirical potential overpasses our computing possibilities. The group of Professor Paweł Dłużewski from Instytut of Fundamental Technological Research of PAS gave us access to the utilities permitting the analysis of such a large system of dislocations by using continuous theory of dislocations and starting from experimental  $\alpha$  fields.

In the experimental work [H5], a similar procedure to the one used in the case of the network of misfit dislocations was applied for the CdZnTe/GaAs interface [11]. The distribution of DDT that I determined was introduced as initial values to the nodes of the finite element mesh for defining the geometry of the problem. Subsequently, the calculations were performed taking into account the anisotropy of the material and nonlinear dependence between deformation and stress for large deformations. The reduction of the stiffness of material was applied to the core region; this is justified by the fact that angles between bonds as well as distances between the atoms differ from the perfect crystal. It is hard to expect that the elastic properties of the core are the same as for perfect crystal. The detailed description and justification of such an approach is given in the works [10,11].

The calculation was performed assuming the threshold condition corresponding to material bulk thickness. This means that we assumed the flat deformation model. No displacement was allowed along the Z direction (corresponding to the propagation of the electron beam) and periodic in the x and y directions. The iterative procedure starting from experimental distortion measured in the thin foil will reproduce the strain condition similar to the one which was obtained in the epitaxial layer before thinning for electron transparency. In this way, we can calculate the  $\sigma_{zz}$  stress field component. This opens up the possibility to bypass one of the limitations of transmission electron microscopy: the loss of information during electron beam direction as well as the reconstruction of the stress field in the step prior to the thinning process. A detailed description of the finite element method was given in the work [25].

The results of my calculations in the work [H5] show that core structures of strongly interacting dislocations are far from the equilibrium state. This means that theoretical calculations of electronic properties for edge dislocation type  $\vec{a}$  should take into account the strong deformation of the cores. On the other hand, the density of dislocations achievable in the active region of devices is many times lower than the dislocation configuration presented there. It seems that for isolated dislocations, the core structure is nearest to the equilibrium state. However, it is also possible that the structure of the cores can also be disturbed not only by stress, but also by doping. It may be possible, in such a way, to find some discrepancy in the results of measurement in the cathodoluminescence of edge dislocation given for example in work [14]. The aspect of core imperfection has not been investigated so far from the point of view of modeling of the electrical properties in nitride semiconductors.

Information about the doping of dislocation cores in an indirect way can be obtained by measuring the potential/charge related with the dislocation core with the use of electron holography. Up to present, there are only a few reports concerning such types of measurement and there is some discrepancy in the results. GaN is a piezoelectric material, so due to strong asymmetric deformation of imperfect dislocation near the core, we can expect strong electrical fields which may disturb the measurement of potential related to the core atomic configuration.

In work [H6] we check if this effect is important for holography measurement. In holography experiment dislocation line is slightly deviated from zone axis by crystal rotation to avoid the channeling conditions [26]. In HRTEM images dislocation line is parallel to electron beam, in both cases from point of view of relaxation of thin foil and influence of free surface are similar.

The experimentally measured distortion fields on the HRTEM image around the edge dislocation can be used to determine the potential related to the piezoelectric field. In our work [H6], we present constitutive equations linking displacements and the electric field, which can be solved numerically in the finite element mesh. As in preceding works, experimental lattice distortions for a network of dislocations are taken as starting values.

The approach discussed in work [H6] was probably the first attempt to determine the piezoelectric field at the nanoscale with the use of HRTEM microscopy. We showed that the electric field related to the dislocation line can reach a value of +/- 8kV cm<sup>-1</sup>. In this example, the calculated potential was very small +/- 0.012V compared to -3V measured in holographic

experiments [26]. However, this was significant if compared with the 0.1 V measured with scanning surface potential microscopy (SSPM) in magnesium and silicon doped GaN[27] samples. It appears that from the point of view of physical properties, the problem of dislocation charge is still unsolved.

#### 4.1.3 Chemical nanoanalysis of coherent heterostructures

The resolution of spectroscopic methods such as EDS, EELS, which are traditionally used for chemical analysis, is limited mainly by the size of electron probe but also by S/N ratio and sensibility of the sample to the electron irradiation. However, the new probe corrected microscopes allow achieving today electron probe size diameters in the subnanometric range. But even now, mapping large area of MQW 50nm x 50 nm with near atomic resolution is still a challenge even for the latest generation microscopes and detectors. Additionally spectroscopic methods required exposition of the sample for high dose of energetic electrons.

In the case of ternary coherent semiconductors, such as nano structures like Quantum Wells (QW) and quantum dots (QDs), containing the active region of a laser diode (LD), it is possible to indirectly evaluate the local composition by measurement of the local lattice parameter on HRTEM images of their cross section by using a relatively low electron dose.

The active region of the GaN based LED and lasers diodes consist of InGaN/GaN multi quantum wells where the recombination of the carriers produces light emission. In real devices, this region also includes GaN epilayers working as light waveguides and AIN electron blocking layers. Part of the region need to be doped with Mg and Si, respectively, in order to form a p-n junction. The quality of this active region determines the main parameters of the devices. The emitted light wavelength depends of the indium concentration and widths of the QW and barriers. But also the quantum Quantum-confined StarkEeffect( QCSE) related to spontaneous and piezoelectric field impact the wave length (read shift) emitted by devices grown in polar [00001] orientation especially for low indium content and QW width over 3nm [28].

Additionally, the potential fluctuation related to the QW width variation and compositional heterogeneousness complicates understanding the luminescent properties of devices. The internal quantum efficiency depends strongly on the structural perfection of the active MQW region, where differences of individual QWs will result in widening of emission peaks. The mechanisms responsible for high performance of light emission in nitride based devices over the last 10 years was a subject of scientific discussion and even today all aspects of this phenomena are not completely clear, especially in relation to structural properties. The high efficiency of the early GaN based LEDs fabricated from layers with huge densities of threading dislocations >  $10^{10}$  cm<sup>-2</sup> [29] started a controversy and debate concerning the mechanism of the carrier localization in InGaN/GaN QWs.

Presently, most researchers agree on the basis of theoretical calculations as well as optical measurements that the emission from InGaN/InGaN MQW is related with highly localized excitonic effects. This localization in QW is in the range of 1-3 nm. However, it is not yet clear what the origin of the localization of the holes and electrons preventing them from drifting to dislocations and performing non-radiative recombination is. One of the concepts is localization of the holes at the atomic level on the chain of atoms In-N-In-N [30, 31]. The existence of such chains is statistically possible in homogenous alloy. So no visible structural variation is expected. This mechanism seems to properly explain the properties of the QW

containing less than 15% of indium. However, another conception exists which assumes that electrons and holes are not localized in the same place [32].

We are still dealing with the model of QW with higher indium content is localization of excitons on indium reach nanometric size clasters. This concept is based on strong arguments: theoretical thermodynamic calculation shows that InGaN compound is instable in grow temperature and indium concentration higher then 20 % of indium [33]. However, this may not be so in the case of the biaxialy strained epitaxial layer [34]. The photoluminescence investigations of InGaN QW show a characteristic S-shaped shift of the spectrum in the temperature function [35], related with the fluctuation of potential in QW of different depth, and is frequently linked with the presence of indium concentration fluctuation

The concept of nanometric indium rich clusters, which localize excitons, was suggested at the beginning on the basis of TEM investigations [36] and was confirmed by many other research groups. However, today it is the object of controversy and debate. The development of this aspect will be presented in the following part concerning a description of the results of the papers [H7-H9]. The potential fluctuation n of the Quantum well can also be caused by other reasons, for example, variation of well width [37]. Changing of the QW width containing 25% indium by 1 ML results in a shift of the PL spectra peek by 58 meV [38]. Structural inhomogenites on the broader scale, such as in big fluctuations of QW width by more than 10 nm distance [39, H12] as well as extended indium segregation on the micrometer scale [40] can effectively screen dislocations and block free movements of the carriers in the structure.

Recently, some authors concluded that more than one mechanism is responsible for effective light emissions from InGaN QW. It seems that from the structural point of view, the drop of efficiency in such a structure in the case of strong driving current can be related to limited numbers of exciton localization centers [41]. By introducing well controlled structural inhomogenites, it may be possible to create a higher density for such a place and break this limitation. The optimal structure for maximum efficiency and for wider wave-length range emissions is still an open scientific and technological problem for this kind of material.

Therefore, the precision determination of the structural properties of MQW seems to be a key issue. Application of the quantitative HRTEM for the analysis of the active region of the LED and LD will permit determining many crucial parameters:

- The geometry of individual QW: width, composition, asymmetry of compositional profiles i.e. segregation coefficient,
- long range compositional lateral fluctuations,
- long range fluctuations of QW widths,
- interface state: interfusion and roughness,
- detection of short range indium segregation or indium reach clusters,
- homogeneity of MQW, repetition of chemical profiles in a QW stack,
- the structural quality of the remaining elements of active regions (light guide, electron blocking layer, mirrors, doping related defects, etc.).

Most of the listed parameters can be obtained directly from TEM images to obtain an overview of all complex structures in comparison to X-ray diffraction, where the simulations and test samples are necessary. In particular, it is possible to quickly obtain a visualization of any geometrical and chemical in-homogeneity of the structure by diffraction contrast, Z-contrast, or

HRTEM on a few micron scales. However, if the quantification of these parameters is necessary, a considerable effort needs to be made for correct application of the technique as well as for understanding all factors which can influence the image formation for this non-centrosymmetric material system with highly anisotropic physical properties.

As to what concerns the application of TEM in determining the subtle features of QWs as subnanometric indium segregation and determining the structure of interfaces, there is still much controversy directly related with the poorly known sensitivity of the compounds and alloys with electron beam damage and differences in experimental conditions of various laboratories. The analysis of small objects nearly 1-2nm diameter is also related with averaging and projection problems in TEM investigations and the quality of ultrathin cross-sections under 10 nm thickness.

Part of my activity for the IPAM project was the measurement of the indium distribution in InGaN/GaN MQW using the strain measurements on HRTEM images. Measuring this composition precisely and reconstructing the real absolute compositional profiles need more sophisticated approaches as compared to the description in the work from the year 2001 [3]. Indeed, many effects should be taken into consideration:

- 1. InGaN is a highly beam sensitive material, so electron dose reduction techniques must be applied.
- 2. InGaN is a non-centrosymmetric crystal system; therefore, the image formation process is complex and information transfer about lattice deformation may be transformed and delocalized in a complicated way. The electron scattering process in crystal as well as deformation of information introduced by the imperfect optical system of the microscope should be taken into account.
- 3. InGaN is a highly anisotropic and piezoelectric crystal: the free surfaces of the thin TEM cross-section due to partial relaxation modify the extracted chemical profile of the QW in a non-homogenous way.
- 4. In the case of compositional fluctuations, only the thinnest part of the cross-section can be analyzed with confidence due to averaging information from the projected image of the sample.

Papers [H7] and [H8] are devoted to the application of strain measurement techniques on HRTEM images to measure the compositions of MQW grown using MOVPE and MBE techniques. The InGaN material is suspected to have a spinodal decomposition when heated [33]; therefore, ion milling was performed with the samples kept at the temperature of liquid nitrogen. High resolution microscopy observations were carried out on a Topcon 002B instrument operating at 200 kV equipped with an LaB<sub>6</sub> electron gun and providing a 0.18 nm point to point resolution. HRTEM [11-20] zone axis images were formed by using many beam conditions. 0001, 0002, 0004, 1-100, 1-102 type beams contributed to the image formation process. For this kind of zone axis, simulations show that the image contrast is very sensitive to foil thickness variation, which help us to make thickness estimation of the observed area with an accuracy of  $\Delta t=5$ nm. The measurement of the local displacement field was performed using the peak finding methods, with the contributions of all beams forming images taken into account, meaning without a high frequency cutting filter.

In order to take into account the thin foil relaxation effects, 2D finite element (FEM) simulations were performed for realistic geometry of the observed samples. The anisotropy and

the nonlinear behavior of the material were included in the FEM algorithm. The model of the thin cross-section reproduced the widths of the wells and barrier and the thickness of the foil. The boundary condition was chosen to reproduce the real strain field in a TEM sample. The calculation was carried out on the  $xy^{\ddagger}$  plane; however, it was necessary to take into account additional deformations along the z direction. In this direction, the lattices of GaN and InGaN are coherent with common lattice parameter  $a_z$ , which depended on the relative thickness of the substrate, QW and barrier widths, chemical composition and elastic constants.

Finally, the distortion component  $\varepsilon_{xx}$  measured experimentally on HRTEM images was converted to composition by a simple expression  $x_{In}=A\varepsilon_{xx}$  where A was determined by FEM modeling and depended only on the foil thickness. Please note the fact that the GaN barrier region in the neighborhood of QW, used as reference region, is under slight compression along 0002 direction, was also taken into account. My primary contribution to the work of [H8] was to propose the concept of determining the absolute indium content based on FEM modeling of strain distribution in the TEM cross-section of heterostructures. I proposed the use of the boundary condition reproducing TEM sample strain state in the development of this scaling concept based on the geometry of the thin foil cross-section model. However, FEM modeling and final implementation of boundary conditions were performed by Dr. G. Maciejewski and Prof. dr. hab Piotr Dłużewski from IFTR-PAS.

When applying this scaling procedure to the MBE sample assuming a 10 nm thin foil, we obtained the elemental 2D distribution of indium in xz plane. The maximum indium concentration was in the range of 27 at%, which was much higher than the expected nominal composition of 15-17at%. The averaged composition profiles for three QW clearly show that the QW in the middle had a lower concentration of indium at about 2-3 at%. From the integrated averaged profiles, it was possible to determine the total indium content in the individual well expressed in ML of pure InN. When the average indium content was calculated, the obtained average values of 16 at%, 13.5 at% and 16 at% corresponded to the nominal indium content range and such obtained parameters do not depend on the shape of the chemical profile.

Strain/composition scaling was performed assuming a homogenous distribution of indium inside QW. In the case of the MOVPE sample, it was clear that we had indium clustering already visible on the HRTEM image. By applying the scaling procedure, the homogenous indium distribution assumed values between 22 at% to 45 at%. If we have indium reaching clusters inside a matrix with lower indium content, it is interesting to estimate the composition and sizes of the cluster. The series of the FEM was performed for different cluster size and composition. The average composition of 16 at% was constrained in this modeling and foil thickness 7 nm. As results we were able to conclude that in the case of MOVPE, the observed fluctuation on compositional maps might be related with the presence of only one cluster of 3 nm wide and 2 nm high. In the case of MBE sample even two small clusters of 1.6 nm could be present in such a 7nm thick foil.

<sup>&</sup>lt;sup>\*</sup> In the case of [H8] axis X || grows towards [0001], axis Y grows towards || electron beam and axis Z|| towards the quantum well, the thickness of foil t is measured long the Y axis.

Therefore, the work [H8] presents a method for determining the absolute concentration of indium in quantum wells. It has been proven that it is possible to determine the structural differences between the individual wells. Work [H8] also shows that there are clear differences in the observed distribution of indium between the MOVPE and MBE samples with a similar nominal concentration, studied under similar conditions. This distribution in the case of MOVPE samples evidently takes the form of indium-rich clusters. In the case of the MBE sample, detected fluctuations can also be associated with the existence of small clusters of indium on unformed shapes. It is important to underline here that thin sections with a thickness below 10 nm have been studied, but this is not sufficient to uniquely determine the shape of the segregations of 0.5-1 nm in size due to the effect of projection.

We must note that the indium clusters observed in the TEM are still subjects of controversy and debate. Gerthsen et al. reported observation of clusters containing 80% indium [42,43]. Later, it was shown by Smeeton et al. [44] that this high strain fluctuation in the QW area can result from electron beam irradiation and formation of "false clusters," particularly in high current conditions of field emission guns (FEG). However, for similar current condition, another experiment performed in the D. Gerthsen group [45] showed that significant change of the QW structure starts only after 2 minutes of irradiation. In our case, when the LaB<sub>6</sub> gun was used (10x less intensity), modification of the structure was relatively long. Today, the formation of the indium cluster during growth is still claimed by the Berkley group [46].

The authors from this group report the negligible impact of the electron beam on the observed structures for up to 2 minutes of irradiation and current densities of 20-40 A/cm<sup>2</sup>. In their paper in 2006 [47], they reported that green emitting In<sub>x</sub>Ga<sub>1-x</sub>N quantum wells (with an average indium fraction x of about 0.2) exhibited genuine indium-rich clusters, 1–3nm wide, with indium content up to 0.40.

In a recent experiment performed in Cambridge, samples of irradiated and non-irradiated MQW were studied by atom probe tomography (APT), and structural differences between the reference and the irradiated sample were detected by S.E. Bennett et al. [48]. However, observed variation in indium distribution was small in comparison to the overall damage caused by 64 minutes of irradiation by the electron beam. It should be noted that a 2 minute irradiation is sufficient to perform a focal series of HRTEM images.

The work of the Berkeley group, and my own research, was performed using very thin areas with a thickness below 10 nm. Research and conclusions by Smeeeton et al. [44] concerning "false cluster" formation have been performed in two beam conditions and relatively thick samples – around 20 nm. As the authors themselves admit, they are not able to detect fluctuations of indium composition on the scale of 1-3 nm due to the effect of projection. At the same time, I emphasize that everybody observes the structural changes in the InGaN wells with prolonged exposure. So, the debate concerning the presence or lack of nanometric size indium rich clusters has not yet been concluded to date. My comment on the subject and proposition of the mechanism responsible for this process is presented in [H11].

Independent of the origin, nanometric scale fluctuations of the measured distortion are present in experimental images of the MOVPE sample presented in work [H7] and [H8]. The

understanding of how such fluctuations reflect real 3D atomic arrangement can lead us to determine the origin of such structures.

Paper [H9] to a great extent is devoted to describing of the 3D Finite Element Modeling of the TEM cross-section by the QW containing burned indium rich clusters with the use of nonlinear mechanics. These calculations were performed by Dr. Grzegorz Jurczak and Dr. Grzegorz Maciejewski at the Material Computation Group under the direction of Professor Paweł Dłużewski. However, this work contains my valuable contribution consisting of the global concept of application of the simulation and image processing sequence for explaining measurements from experimental HRTEM image distortion maps showing strong fluctuation in the QW region. I also proposed the geometric model of burned clusters and performed: image processing of experimental and simulated images, generation of the atom position in supercells on the basis of 3D FEM results and image simulations. I presented conclusions related to the overall approach based on a comparison of the iteratively improved model of the structure with experimental data and the impact of dynamic scattering and image formation process under microscope, which described the deformation and delocalization of information concerning distortion on obtained maps.

The proposed in paper [H9] simulation approach takes into account most of the effects which can influence the distortion measurement on HRTEM by reproducing a real word condition of strain relaxation of a thin cross section containing the indium reach cluster taking into account the anisotropy and nonlinear behavior of material under strong deformation. The **dynamic effect of electron scattering corresponding to multiple beam conditions of HRTEM image formation was taken into account**. It was proved that the distortion field measured on a simple projection of a supercell differs from distortion measured on HRTEM image simulations. But these differences for an optimized condition and thin cross-section < 10 nm for many beam zone axis images were not overcome  $\Delta \varepsilon + /-0.004$ , and the shape and size error of the cluster measured at HWHM did not exceed 0.3 nm. The results presented in paper [H9] confirm the existence of 2 to 3 nm of burned indium rich cluster in QW of investigated MOVPE samples.

In HRTEM images, the position of maximum contrast is determined by constructive interference of all beams or at last 2 beams, which pass through objective aperture. In papers [H7-H9] the peak finding algorithm was used to determine the distortion field [3]. In paper [H10], the Geometric Phase Analysis (GPA) is applied to study the influence of the imaging parameters based on the reliability of the reconstruction of the real compositional profile from HRTEM images obtained in the zone axis in multi-beam conditions. In the case of GPA, one or two lattice fringes (reflected beams) are used to construct the distortion field. In paper [H10], the difference between theoretical and extracted from simulated HRTEM images distortion profiles was analyzed with the use of different sets of lattice fringes.

We analyzed this using the thin TEM cross-section model of the InGaN QW with a rectangular compositional profile of indium concentration. Atom positions in the supercell were determined according to FEM modeling for thin foil relaxation. A series of simulated images obtained by the multislide metode for the condition corresponding to the Topcon 002B microscope were analyzed with the GPA.

The paper concludes that the direct peak-finding procedure is more suitable for analysis of such zone axis HRTEM images as compared to the GPA method. This is due to fact that for

the non-centrosymetric zone  $axis^{\$}$ , the information about distortion of the lattice is distributed between the beams in a very complicated way. Additionally, such information is modified by the contrast transfer function CTF of the microscope. The results presented in papers [H9] and [H10] show that only using a complete set of information from all beams, a reliable profile can be reproduced, as in the case of the PF algorithm. A more detail analysis and explanation is given in the following paper [H11].

In papers [H9] and [H10] for interpretation and validation of the experimental distortion/concentration maps the approach based on "real word simulation " of thin TEM crossection model of homogenous QW with rectangular compositional profile as well burned in QW indium reach clusters was used. I showed that it is possible to precisely determine chemical indium distribution inside the QW with segregation on the basis of the multi-beam HRTEM images obtained in axial illumination conditions. I determined the limits and errors of such measurements. This work concluded my activity and interpretation of results obtained in the CIMAP laboratory using a TOPCON 002B microscope for the MQW of InGaN/GaN.

After my return to IPPAS, part of my scientific activity was devoted to continuing the investigation of quantum nitride based heterostructures. Part of this investigation was performed on UNIPRESS samples in the framework of the project [Pro8] by Dr. Grzegorz Maciejewski from IFTR PAS. The transmission electron microscope installed in IPPAS JEOL 2000EX has lower performance as compared to the Topcon 002B. The main difference between these two types of microscopes is the spherical aberration coefficient and point resolution, which are in the order of 1.7-1.8 mm for Jeol 2000EX and 0.4 mm for Topcon 002B with respective point resolution of 0.27 nm and 0.19nm.

The limited resolution of our microscope installed in the IP-PAS excluded an investigation of InGaN/GaN heterostructures under zone axis illumination conditions. The 0002 lattice planes in the GaN are separated by 0.25 nm, which exceeded the point resolution of microscope Jeol 2000EX.

In paper [H11], I solved this problem by using lattice fringe images in **non-axial illumination conditions**. Observations of MQW were performed on the [11-20] and [1-100] zone axis. Images of 0002 lattice fringes were obtained by a sample tilting of  $3^{\circ}$ –  $4^{\circ}$  around the [0001] direction away from the exact pool position. The incident electron beam was inclined from the optical axis of the microscope at the half angle between the 0002 and 0000 incident beams.

These conditions are optimal for Jeol 2000EX microscope but different from those suggested in paper [49] for chemical composition mapping, where the 0002 beam is placed on the optical axis. The positioning of the 0002 beam on the optical axis results in the incident beam being at the border of the diaphragm and in a temporary envelope of the contrast transfer function (CTF) and information limit of our microscope. As a result, the fringe contrast as well as its S/N (signal-to-noise) ratio was weak due to the damping of the incident beam.

We verified the validity of our choice by image simulation for the rectangular compositional profile of indium. This simulation was analogical to the ones describe in paper [H10] and takes into account the column bending effect and realistic propagation of the electron

<sup>§</sup> This mean that in diffraction pattern, the phase and amplitude of the g reflex is different then -g.

wave by a deformed crystal. Results of such image processing of simulated fringes images are summarized in fig 1b [H11].

The observable position of the QW is a function of defocus. However, the maximum strain level and the QW width do not change with the defocus. The imaging process does not introduce significant additional broadening of the interfaces, but the FWHM is smaller by 0.4 nm compared to the FEM profile near to the starting rectangular profile of indium concentration. Performed validation of chosen imaging conditions prove that reliable determination of the indium composition profile is possible by using such a microscope.

Structural perfection of MQW is critical for the performance of light emitting devices. Paper [H11] showed how to improve the Q-HRTEM (quantitative –HRTEM) methodology in order to analyze the entire active InGaN/GaN region. It contained 10 QW and measured 220x220 nm. Keeping a constant condition of image formation in phase contrast for a large field of view is a difficult task mainly due to foil thickness, sample Z microscope position and orientation variations.

To make possible comparison between all 10 QWs, the effect of thin foil thickness variation should be resolved. In paper [H11], the local foil thickness is measured using "thickness fringes" methods. Images obtained in dual beam non-axial illumination conditions with slight deviation from the [11-20] and [1-100] zone axis show well-defined extinction fringes. Bloch wave simulations performed for GaN crystals for tilt range from 0 to 4.5° show that "thickness fringes" have a constant period starting from 1.5° deviation from the zone axis. Using this information about the position of the maximum and minimum of such fringes, a map of foil thickness was determined. Precision of the thickness measurement is higher than 5nm. Distortion measured by the GPA was used to construct an average profile of the indium profile for all QWs, but averaging was performed only for the same foil thickness (in the case of fringe image, all information about distortions is stored in 0002 refraction so the GPA can be used).

For known foil thickness, the absolute indium concentration was obtained from strain mapping similar to work [H10]. Next, we established the maximum and average indium composition, total indium content segregation coefficient, and QW width using different criteria for each QW separately. The average value for all 10 QW was compared with the value determined by high-resolution X-ray diffractometry.

Results presented in work [H11] are one of the few reports of direct comparison of absolute values of indium concentration as well as parameters of InGaN/GaN MQW obtained by systematic quantitative QHRTEM analysis, including the results of X-ray diffractometry (XRD). Agreement was reached between the absolute average indium concentrations (quantum barriers and wells) determined by TEM and XRD independently. The difference of absolute indium concentration varied from  $\Delta x=0.001$  to 0.008 (for the values at the level from 0.027 to 0.06 respectively). Differences of period determinations were at the level of 0.5 nm. It should be noted that only these values are directly obtained from X-ray diffractometry (XRD) measurement. The remaining parameters such as QW and QB width and QW indium concentration are the result of simulation and fitting of experimental curves, which need introduction to additional information concerning the ratio of the thickness between the barrier and well.

In TEM investigations, such information is directly accessible. It is possible that the observed differences can be related to different sampling sizes in TEM and XRD investigations and the heterogeneousness of the samples on a larger scale. The approach proposed in work [H11], which takes into account only regions of the TEM sample with the same thickness, has very high relative precision and permits quantitative comparison of QW in the MQW stack. In literature, there are only a few quantitative studies of the whole MQW by TEM, and my results represent the biggest ever analyzed field of view. The differences between individual QWs of the investigated samples are small compared to those reported in the literature [50], where the variation in foil thickness seems to be neglected.

Paper [H11] also reported the results of irradiation of high indium content MQW as contributing to the debate concerning the presence of the rich in indium clusters. The observed contrast changes induced by the electron beam cannot be explained by the formation of clusters similar to those reported in papers [H7 and H8], where the overall HRTEM image quality was very high and clusters were observed at the beginning of the electron-beam exposure. A possible explanation for the strong diffraction contrast is that some of the indium atoms diffuse to the surface of the thin foil and form small indium-rich islands. This produces undulation in the foil, strongly influencing the diffraction contrast. However, this effect is minimized by averaging the surface signal with the internal part of the QW and, as such, does not affect the maps of the  $\varepsilon_{0002}$  strain component.

The methodology of composition measurement with the use of lattice fringe images developed for the Jeol 2000EX microscope was applied to MQW grown by MOVPE in two temperature processes in work [H12]. In this case, the individual fluctuations of the width and composition variation are in the 30-120 nm range. This required analysis for the greater fields of view than in the case of paper [H11]. This work was performed within the European RAINBOW project Marie Curie Initial Training Networks (ITN) "High Quality Material and Intrinsic Properties of InN and Indium Rich Nitride Alloys" in which I was coordinator of the work for IPPAS in the framework of consortium 14 industrial and Academic Partners.

The high electron beam sensitivity of InGaN with a high indium content makes necessary the limitation of the total electron dose. All analyses presented in paper [H12] are based on the first images performed during the first 10s of irradiation. The investigated MQW showed significant structural modification on the nanoometric scale, and occurred in a few minutes. In order to obtain the thin cross-section, which at a distance of 200-300 nm foil thickness the variations do not exceed 5 nm, I proposed and improved the ion milling procedure by applying LN2 cooling and progressive variation of ion energy.

Mapping of indium concentration clearly shows that during the dual temperature process for the rich indium layer, the unprotected InGaN layer during the temperature ramp necessary for GaN growth becomes degraded, probably by desorption of indium. The 1 to 4 monolayer high steps are created on the second surface of QW. Such roughness is partially removed during grown of the GaN barrier. However, in latter grown, the QW's bottom surface also becomes rough. In some cases, the QW are almost interrupted where the QW width drops to 2MIL and indium concentration to 5 at% from an average of 20 at%.

The observed roughness of QWs in the MQW cross section in [11-20] and [1-100] zone axes shows anisotropy of morphology. This can mean that diversity of the QW width can have stripe like morphology. The plan view diffraction contrast images performed in the [0001] zone axis shows stripe like structures. In paper [H12], we apply the Fourier transform method to determine the anisotropy of such stripe structures. From the maximum amplitude of the FFT, the lateral characteristic dimension (LCD) of the contrast features is determined, which correlate quite well with our cross-sectional images.

These structural non-homogeneities can have a strong impact on the carrier recombination as suggested in [39] for similar structures. The steps of QW interfaces can act as localization centers and improve efficiency, but can also spread peeks PF of spectrum.

Findings described in paper [H12] inspirited research to discover a way to protect the high indium content layer from desorption by using the thin GaN protective layer growth at the temperature of InGaN growth. The analysis of such an MQW was performed under my supervision by Ph.D student Francesco Ivaldi. The results of this work were presented in paper [51]. In this paper, we showed that for samples with protective layer the PL spectrum peek positions have a green shift as compared to unprotected InGaN QW. The result is coherent with the quantitative –HRTEM (QHRTEM) analysis, which clearly shows more indium in QW as well as more homogenous well width when the LT GaN protective layer is applied.

On the other hand, we showed that even then the crystal quality of the low temperature LT-GaN protective layer is good. The periodicity of the MQW with application of the LT-GaN protective layer is worse compared to reference samples, and this is consistent with the XRD data. I want to underline that data concerning the MQW structure with broad QW width fluctuations presented in papers [H12] and [51] is difficult to obtain by analysis of the X-ray spectrum details. Actually, only atom probe tomography ATP and AFM permitted obtaining direct information with sufficient spatial resolution concerning the lateral (in the xy plane) structure of QWs. Such methodology for chemical mapping was applied in the following works in the framework of the RAIBOW project, not only in IPPAS but also in CIMAP [52] for analysis of indium containing nitride heterostructures.

The method of strain measurement on HRTEM images and was applied in work [H13] devoted to analysis of the axial ZnTe/CdTe nanowires (NW) grown by MBE in the VLS (Vapour Liquid Solid) mechanism with the use the gold-based catalyst. My contribution to this work consisted in measurement of the {111} lattice fringes spacing on HRTEM mages taken in <112>

zone axis in the transition zone between ZnTe and CdTe parts of the nanowires. The interface width determined by this method is 56 nm. This relatively large width is caused by the use of the VLS growth method in combination with the MBE technique, where crystallization from the liquid phase occurs and replacement of Zn atoms by Cd atoms is not immediate. Our results are an important step toward the realization of CdTe quantum dots inside ZnTe NW. Such nanowire-based quantum dot structures can find their applications as single photons on demand for sources in the future. Information about the width of the transition zone permitted further works in IPPAS to take place with optimum growth conditions and minimizing the width of this transition zone. As a result, there was successful realization of the formation of optically active CdTe quantum dots in ZnTe nanowires in 2011 [53].

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## 5. Other scientific achievements

## 5.1 Achievements before obtaining a doctoral degree

#### 5.1.1 Master's thesis

The subject of my master's thesis was an investigation of the deposition process as well as of the structure of heat resistance coating based on multi-compound nickel alloys fabricated by the pulse plasma method. Optimization of the deposition process included magneto-hydrodynamic simulation of the co-axial plasma gun; long-term resistivity was also part of this work. However, the main part of the work was focused on structural investigations of the coatings and structural modifications during annealing–oxidation cycles. In these investigations optical microscopy, scanning electron microscopy, powder X-ray diffraction and Wavelength Dispersive X-ray techniques were used to determine the phase content of this multi-compound material. During the last year of my studies at the Warsaw University of Technology I obtained a fellowship funded by the Scientific and Production Center in Warsaw (CEMI).

## 5.1.2 Work in the semiconductor industry

After obtaining my master's degree in 1989 I started work at CEMI in the department of development and technology of basic electronic components. I was responsible for the technology and production of power transistors with mesa processing. I actively participated in group work responsible for the development of new processes and production implementation of high-voltage planar transistors in Darlington pairs with triple diffusion for automotive ignition applications. My activities included the design of lithographic masks and contribution to the development of diffusion processes on the production line. This work was finished with test series of this new generation of transistors which had never been fabricated before in Poland before the factory was liquidated in 1993 (due to the economic transformation in Poland during this period).

## 5.1.3 PhD Thesis

In March of 1993 I started my doctoral studies at the Institute of Physics PAS and at the Université Pierre et Marie Curie PARIS 6. This was possible due to a fellowship from the Ministère de la Recherche of the French government in the framework of a Polish-French collaboration program between Groupe de Physique de Solides, Universités Paris VII et Paris VI, Laboratoire de Physique du Solide Ecole Supérieure de Physique et de Chimie Industrielles de la ville de Paris (LPS-ESPCI), IPPAN and UNIPRESS. I started work on my thesis in a newly built MBE laboratory under the direction of Prof. dr hab. Jacek Kossut. My activity was mainly devoted to measuring in-situ lattice relaxation during the MBE of strained CdTe/ZnTe and GaAs/CdTe heterostructures. This measurement was possible due to a modification of the software designed for RHEED oscillation measurements.

The modifications I made permitted the precise determination of the "streak" distances on RHEED video images and the observation of lattice parameters in-situ during growth. This measurement of the dynamics of lattice relaxation during the growth of ZnTe on CdTe was presented at the Jaszowiec Congress [pk1] and published afterwards [p87]. This publication resulted with the collaboration with dr Ferenc Riesz from Hungarian Academy of Sciences,

Research Institute for Technical Physics and Materials Science and paper where the results were confronted with the model of relaxation [P85, PK6].

While preparing my PhD thesis, I was working in close collaboration with the Electron Microscopy Group of IP PAS under the supervision of Dr Elżbieta Mizera, where I started TEM investigation. My participation in the Autumn School of Electron Microscopy in Halle in Germany directly before my departure for France inspired me and focused my interest on quantitative electron microscopy. During my stay in LPS-ESPCI in Paris I performed HRTEM investigations of the self assembled InGaAs QDs from CRHEA(Centre de Recherche sur l'Hétéro-Epitaxie et ses applications) laboratory in France and CdMnTe QW obtained by MBE using "digital alloying" method from IPPAS in Warsaw.

This research was carried out under the supervision of Dr Claude Delamarre and DR J-Y Laval with the collaboration of Dr Genevieve Schiffmacher in the field of HRTEM image simulation. Microscopy observations were performed using a Philips CM20 microscope installed at the Ecole Centrale near Paris with the kind permission of Prof. Bernard Jouffrey. At this time I created software from scratch which permitted the quantitative analysis of high-resolution images for the determination of local lattice distortions using a peak-finding algorithm.

By using this software I determined 2D maps of lattice distortions for epitaxial layers of InGaAs on GaAs substrates. I demonstrated that, due to the high energy of dislocation creation in this system, stress relaxation can be carried out initially without the creation of a defect but by the growth of coherent islands – QDs according to the Stranski-Kranstanow mechanism. The results of this investigation were presented in 1996 at the congress of Réseau Francilien de Microscopie Electronique in Paris [pk5] and during 6 following meetings [pk7, pk8, pk9, pk11, pk12, pk13] including one invited presentation as well as additional publications [p82, p83, p84]. This was the first experimental 2D mapping of elastically relaxed, uncapped, self-organized InGaAs islands showing lattice deformation in a parallel and perpendicular direction to interface<sup>\*</sup>.

My experimental maps were in good agreement with the distortion distribution predicted by the theoretical models of this type of elastic relaxation made possible by bending the {022} lattice planes. These results were important from the point of view of understanding the growth and optical properties of the QDs based on the InGaAs system.

During this period, under the supervision of DR J-Y Laval, I also carried out an analysis of the interfaces and structure of YBaCuO/PrBaCuO superconductive epitaxial layers [pk2, pk3, pk4]. Together with Dr Henri Souchay from the Ecole Centrale, I started using the Geometric Phase Analysis (GPA) of HRTEM images. This collaboration was reported in conference presentations [pk10, pk14].

While preparing my PhD I also investigated the "Digital Magnetic Quantum Wells" grown at IPPAS by the MBE method. The application of high-resolution electron microscopy permitted me to prove that it is possible to grow, by MBE, super lattices of CdMnTe/CdTe with clearly defined interfaces even when the individual QW width was 2 to 6 monolayer thick [p86]. These results confirmed the efficiency of the growth methods and explained the observed magnetic properties of this type of QWs.

<sup>\*</sup> Earlier, Tilman et al. published their results on the one-dimensional measurement of {002} distances of lattice fringes for similar InGaAs islands, but the distortion in direction {022} was neglected. K. Tillman, D. Gerthsen, P. Pfundstein, A. Forster, and K. Urban, J. Appl. Phys. **78**, 3824 ~1995.

# 5.2 Scientific achievements in other than habilitation research subjects after obtaining a PhD degree.

After defending my PhD in Paris in May of 1998 I was employed at IPPAS as a physicist and then as an assistant in the Electron Microscopy Group in the Laboratory of X-ray and Electron Microscopy Research. I continued analyses of experimental data on self-organized InGaAs islands and CdMnTe QWs with a programmed compositional profile fabricated using the "digital alloying" method, this data had been collected during my stay in France. This material was only partially included in my PhD thesis. The results of a deep analysis of this material were published in paper [p79], in which additional information about the threedimensional distribution of the indium atoms inside uncapped InGaAs islands was achieved by using a combination of the 2D distortion maps and Finite Element Modeling. In this paper our model explained the escape of indium atoms from the borders of islands to the center. In paper [p68] the subject of indium atom distribution in coherent InGaAs islands was developed and our results were confirmed by EDS spectroscopy.

EDS measurements were performed at ESPCI (Paris) using a new microscope with a field emission gun which became operational in 1998 after I had defended my PhD. The analysis of the stress concentration in the contact point of two adjacent InGaAs islands and the resulting creation of defects in this area was published in [p80]. The invited talk at the International Electron Microscopy Congress in Durban [pk36] was devoted to these two topics. I also completed an investigation of the profiled magnetic QWs II-VI and the results of my analysis were presented at [pk15 and pk16] and published in [p78]. This investigation confirmed that by using the "digital alloying" method in MBE it was possible to precisely obtain any defined shape of a compositional profile for the QWs. This was demonstrated on the example of trapezoidal Mn elemental profile in CdTe/CdMnTe QW [K1]. The main results of my thesis were also presented during two seminars in the Faculty of Physics at the University of Warsaw [Se1, Se2].

On July 6, 1999 the Scientific Council of the Institute of Physics of the Polish Academy of Sciences confirmed the equivalency of my PhD degree specialization in "physique des solides" obtained in France with the Polish scientific degree of doctor of physical sciences. In December 1999 I became employed at the IP PAS as an adjunct.

During this time I started collaboration with Prof. dr hab. Paweł Dłużewski from the Institute of Fundamental Technological Research PAS (IFTR PAS) in work related to the application of the continuum theory of dislocations in an HRTEM investigation in the framework of a project devoted to this subject [pro2]. In the year 2000 I commenced my post-doctoral internship in the framework of the European IPAM (*Interface analysis at atomic level and Properties of Advanced Materials*). Collaboration with the IFTR PAS and participation in the IPAM project had a significant impact on the direction of my future scientific activity, which is described in part 4 of the summary of scientific accomplishments.

After returning from my internship I started work carried out in the framework of the European Multimetox project coordinated at the IP PAS by Dr hab. Piotr Dłużewski [pro4]. My contribution consisted in work related to TEM sample preparation [pk38] and analysis of the CoCu alloy structure [pk48,pk50] as well as of the superconductive layers of  $Nd_{0.67}Sr_{0.23}MnO_3/YBa_2Cu_3O_7$  [pk35].

During this period I summarized my knowledge and experience related to the quantitative method of HRTEM image analysis in a review paper [p73] and Chapter 9 of a book entitled "Nitride Semiconductors: Handbook on Materials and Devices" [RK1]. The first publication consisted of a literature review illustrated by my own results as well as partially with material

delivered by Prof. A. Rosenauer and Prof. D. Gerthsen. The book chapter [RK1] was illustrated only with those results which I had taken significant part in and the review was devoted largely to the methods suitable for an investigation of nitride semiconductor heterostructures. I also had the opportunity to present a series of invited lectures on this subject during international conferences and workshops organized in Poland and in Spain in the years 2000–2008 [pk53, pk49, pk66, pk61, pk62, pk77]. All along I also gave seminars at IFTR PAS [se3, se4], IP PAS [se5], Lille University [se6], the Faculty of Physics at the University of Warsaw [se7] and Humboldt University in Berlin [se8].

The Laboratory of X-ray and Electron Microscopy Research at the IP PAS is a place where scientific subjects being carried out in many laboratories, not only those at the Institute of Physics, but also in Warsaw and other European research centers, come together and intersect. This laboratory's main role is the structural characterization of samples coming from collaborating research groups. One of this laboratory's important duties is to develop methods suitable for delivering precise information necessary for the development of technology and an understanding of the materials' properties and structures of current interest. Due to the wide range of subjects dealt with within the Electron Microscopy Group, the following description of my scientific achievements is related only to that part of the work that I participated in and which was documented by publications or conference presentations. Below I present a brief overview of my contribution and its impact on each work and research topic.

#### 5.2.1 Semiconductor quantum structures

Already during my post-doctoral internship I was involved in research on the configurations and correlations of CdTe QDs in the framework of projects [pro1, pro3]. For this purpose I improved the software I had written during my PhD, which allowed an analysis of HRTEM images containing 500000 atomic columns. I also implemented a GPA algorithm which permitted analysis of large configurations of the II-VI heterostructures containing superlattices of small CdZnTe QDs (nanoinsertions) in the ZnTe matrix. My most important achievement related to this subject was the discovery of both the ordering of small CdZnTe QDs in samples where spacer thickness was smaller than 25 ML and the absence of such ordering for thicker spacers. These results are in agreement with the optical investigation, where an exciton localization was observed on paired CdTe islands [pk24, pk26, pk28, pk52, p74, I1]. This discovery was also confirmed by the Small Angle X-ray Scattering (SAXS) experiment.

Additionally, in this investigation we discovered the possibility of creating pseudocrystals by correlated CdTe [p72] nanoinsertions. The superlattices of small QDs were interesting objects for testing our advanced approach for HRTEM interpretation based on computer simulations. In works [K3, pk27] I showed, based on measurement of the lattice parameter of HRTEM images, that such nanoinsertions were not pure CdTe, as was assumed in earlier publications, but contained only 35–80% of Cd in the CdZnTe. The analysis of images obtained along different projections and compared to models of cadmium atom distribution confirmed the hypothesis of diluted CdZnTe QDs [pk41, pk47, pk54]. The equilibrium positions of Cd atoms in the model were calculated with the use of the S-W potential by Dr Piotr Traczykowski at IFTR PAS.

#### SiGe Quantum dots

Part of my scientific activity was devoted to the investigation of self-organized structures on the basis of SiGe. My contribution to this research consisted in determining the chemical composition with the use of distortion analysis on HRTEM cross-section images of QDs. In paper [p45], the determined distribution of Ge and Si in capped QDs is in agreement with the average compositions from the X-ray Absorption Fine-Structure in XAFS measurements (performed by Dr. I. Demczenko and Prof. dr hab. K. Jabłońska) as well as with local EDS measurements (performed by DR J-Y Laval and Ph.d student M. Żak). Information on Ge atom distribution and the QD dimensions allowed us to explain the low energetic part of the experimental spectrum from these kinds of QDs. Paper [p39] was devoted to the development of a method of determining the shape and chemical profile in uncapped self-organized SiGe islands. The plan view images of large coherent SiGe islands, obtained in the dark field, show a characteristic "pseudo moiré" contrast.

My contribution to this work was the development of a simulation method of this kind of diffraction contrast starting from a three-dimensional model of distortion in a SiGe island and the substrate calculated by FEM. An analysis of the series of simulations showed that it is possible to determine the shape of the island (dome or pyramid) and the fact that there can be a homogeneous composition or presence of a compositional gradient. In relation to the HRTEM investigation, the advantage of this method is the possibility of analyzing many QDs in a field of view of a few square microns. In a following work [p21], the structural modification occurring during the capping of SiGe islands of different shapes and sizes was investigated. Analysis by HRTEM, EDS and AFM showed that even a very small layer of Si very strongly disturbs the balance between elastic and surface energy of coherent islands, leading to rapid changes in the morphology of the islands, which in extreme cases leads to the transformation of quantum dots in quantum wells. The results of the SiGe dots were presented at several national and international conferences [pk58, pk67].

## InAs/GaAs natural quantum dots.

I also contributed to investigations of the exciton localization discovered by the  $\mu$ -luminescence method by Dr hab. Adam Babiński in the wetting layer of InAs/GaAs occurring between large self-organized QDs. The lattice distortion analysis which I performed on the HRTEM images obtained by Dr Jolanta Borysiuk clearly showed a few nanometer wide fluctuation of indium concentration inside the InGaAs wetting layer. This discovery permitted the interpretation of  $\mu$ -Pl spectra as a signal coming from "natural quantum dots", at variance with the signal from self-organized QDs [p30, p31, p25, pk90]

## InN and InGaN quantum dots.

In 2008 I started the RAINBOW European project [pro14] (as principal investigator at IP PAS). One of the objectives of the RAINBOW project was the growth of InN and InGaN QDs by MOVPE and their potential application in the fabrication of optoelectronic devices and light emitters in the range of long wavelengths (green, amber) in close collaboration with the research group at Experimental Nanophysics and Photonics at the Technische Universität in Berlin and with AIXTRON SE as the industrial partner.

This investigation was carried out under my supervision by PhD student. Francesco Ivaldi, who was employed by the RAINBOW project. The structural modifications which occurred during the capping of such structures at different temperatures were characterized. In

paper [p6] we analyzed the capping process of InN QDs with low-temperature GaN. Even for this very low temperature (520 °C) of GaN cap deposition, the InN QDs were degraded, thus forming an InGaN wetting layer. Q-HRTEM and X-ray diffraction confirmed the creation of a ternary InGaN alloy. The results of this investigation were published in the Japanese Journal of Applied Physics [P6]. In the case of the capping of self-organized InGaN QDs on a 20% InGaN wetting layer with the GaN grown during the temperature ramp up to the optimal GaN growth temperature, we observed a partial decomposition of the InGaN and creation of the metallic indium precipitations, as confirmed by the presence of the metallic indium plasmon peak in the EELS spectrum[p128]. These results will be the subject of a future publication.

#### Annealed InGaN/GaN multi-quantum wells.

Modifications of the structure occurring during annealing in a temperature range of 880– 975 °C in the MOVPE reactor of InGaN multi-quantum wells (MQWs) grown by ammonia-MBE in Prof. Nicolas Grandjean's EPFL group in Lausanne were investigated at the atomic level. It was concluded that the distribution of indium atoms changes due to different mass transport processes. Moreover, we observed a modification of the morphology of the surface from undulated to flattening. We also investigated the changes of local electronic properties of such quantum wells using Valence Electron Energy-Loss Spectroscopy (VEELS).

Experimental spectra were compared with the results of ab initio calculations performed with WIEN2K software. The results of the investigations were presented at the following international meetings: E-MRS 2011 in Nice [113], MSM-2011 in Cambridge [118], ICNS-9 in Glasgow [125] and the XIVth Int. Conf. on Electron Microscopy in Wisła [119]. They were also published in the Journal of Physics Conference Series [P3]. The analysis of HRTEM images and ab initio calculations was carried out by phd student Francesco Ivaldi, and I carried out all of the experimental observations on the Titan microscope.

## InAs/GaAs i InAs/InP quantum wires and quantum rings

During informal collaboration with Cadiz University in Spain with Prof. Sergio Molina's research group, works related to burned InAs/InP quantum wires [p56] and multilayers of InAs/GaAs quantum discs [p55] were published. The conclusions presented in these papers were based on my analysis of the HRTEM images of structures obtained by the co-authors. These results have been included as an important part in the PhD thesis of Dr Teresa Ben Fernandez [<sup>†</sup>].

<sup>&</sup>lt;sup>††</sup> PhD at Cadiz University 2006 entitled "CONTROL DE LA DIST. Y MORF. DE NANO-HILOS/ANILLOS SEMICOND. III-V AUTOENSAMB. CRECIDOS MEDIANTE MBE" Teresa Ben Fernandez http://www.uca.es/grupos-inv/TEP120/docs-del-grupo/tesis\_dirigidas/737

## 5.2.2 Development of the research methods

#### Algorithms for image analysis

During my two stays at Cadiz University I had the opportunity to collaborate with Dr Pedro Galindo in a subject related to the analysis of HRTEM images. Together we started work on implementing a Peak-Pair algorithm he had proposed based on the affine transformation. What followed work in this subject was a common paper in Ultramicroscopy [p46]. Finally, Dr Pedro Galindo compiled this work by writing commercial software permitting the analysis of distortions on HRTEM images with defects by using the PP algorithm. http://www.hremresearch.com/Eng/plugin/PPAEng.html

#### STEM image correction

I also had the opportunity to remotely participate in works under the first testing and diagnostics of the "Super Stem I" prototype microscope in Daresbury, England. This was a VG HB 501 scanning transmission microscope which was updated by the addition of a new generation of aberration corrector of the condenser system from the NION company. My role was limited to studying the distortion generated by external magnetic fields and the electronics of the microscope and to inventing the procedure for their correction. [p49]. I also participated in an analysis of the first results obtained for GaAs/InGaAs QDs obtained by Dr Ana Sanchez with the use of this prototype microscope [p51].

## Quantitative High-Resolution Transmission Electron Microscopy

The main part of my methodical works has been to take into account the relaxation process in thin foil by applying the Finite Element Method (FEM) as well characteristic parameters of microscopes as detailed in part 4.

#### New TEM methods

Carrying out project [pro15] and the installation of a FEI Titan 80-300 High-Resolution Analytical Microscope at IPPAS in 2010 opened up new investigation possibilities and removed the limitations that had been imposed by the old Jeol200EX microscope. Aberration correction of the objective lenses enables the observation of structures with an atomic resolution, and the monochromated electron beam coupled with a very sensitive EELS detector allows to investigate the local electronic structure. One of the techniques that I developed together with post-doc Dr Angelique Letruit (employed in the framework of the RAINBOW project) was the application of local measurements of the plasmon peak position in the EELS spectra to map the local indium concentration in InAIN. We experimentally determined and correlated with the ab initio calculations the relation between the indium concentration and the maximum position of plasmon energy. The results were presented at the EMRS 2011 congress in Nice [114] and published in [p1].

I am currently developing the methods and exploring the possibilities of our new instrument in the framework of an obtained habilitation grant [pro17]. I have already developed and presented a method of the negative spherical aberration coefficient for the visualization of nitrogen atoms in nitride semiconductor structures. This permits, for example, an analysis of the

polarity in GaN/InGaN [pk120]. I am currently working on developing quantitative scanning transmission electron microscopy using HAADF and BF/DF detectors. I am also testing the possibilities of applying Electron Holography to mapping localized charges in heterostructures (p-n junctions), and together with Dr Marcin Klepka we are developing methods of sample preparation by FIB (Focused Ion Beam) which is suitable for electron holography.

#### 5.2.3 Epitaxial layers

## Spintronic materials: MnAs/GaMnAs, MnSb, silicon implanted with Mn

Another subject is related to the epitaxial layer for spintronic applications. In paper [p53] I determined the structure of self-organized MnAs QDs created during the annealing of GaMnAs covered by amorphous arsenic.

In work [p32] I analyzed the impact of the annealing on the structural modifications in Mn-implanted silicon. Among other things, I showed that after implantation at 610K and annealing at 870K, three zones with different structures are created. In the overshoot area, the stacking faults are the predominant defects, whereas in a second zone with maximum Mn concentration small precipitates (3-6 nm) are observed and, beyond, there is the third zone with {311} plane defects created by the reorganization of knocked out interstitial silicon atoms.

With the support of the Center of Excellence CEPHEUS and thanks to the kindness of Dr Martin Hytch from the CEMES center in Toulouse, in 2005 I had the opportunity to carry out research using the Tecnai G2 F20 S-Twin transmission electron microscope equipped with an aberration corrector of the objective lenses and the GIF – "Gatan Image Filter", which is an energy filter for electrons (it was at that time one of the first microscopes having such possibilities). By using the EF-TEM (Energy-filtered TEM) method I proved that the observed small precipitations were rich in Mn [pk69]. This was confirmed by the EXAFS results performed by Dr Anna Wolska and Prof. Krystyna Ławniczak-Jabłońska [p28, pk68]. My stay at CEMES allowed me to recognize the possibilities and importance of new research techniques (which were unavailable at that time in Poland) and encouraged me to seek opportunities providing such investigations.

In the framework of project [pro11] I was also involved in determining the structure of MnSb/GaAs epitaxial layers. [p22]. Based on TEM studies, I described the shapes, dimensions and structure of MnSb precipitates in the GaMnSb epitaxial layers [p9, p11, pk106].

## 3C-SiC

I also continued investigations of the buffer layers. In the Si/3C-SiC system, lattice mismatch is in the order of 20%. 3C-Sic is a promising material for MOSFET high-power transistors and can also be applied as a substrate for GaN-based structures. Through collaboration with Dr Marcin Zielinski, who is studying the epitaxial growth of the SiC epilayer on silicon substrates at the Novasic company and the CRHEA laboratory in France, I had the opportunity to carry out an investigation of the structure of such layers. In works [p37, p27] we investigated the structural causes of the asymmetric bending of disoriented silicon wafers with several micron-thick layers of 3C-SiC on [001] Si.

The results of the XRD measurements do not show asymmetry in the lattice parameter in the [110] and [1-10] direction. However, TEM observations revealed asymmetry of directional ordering of the stacking faults. By applying non-axial high-resolution microscopy techniques, it

was possible to obtain high quality HRTEM images of the 3C-SiC/Si interface using the Jeol 2000EX microscope. Further analysis of the HRTEM images showed that the distance between the Lomer dislocations on the flat parts of the steps is from 1.4 to 1.5 nm, so this is very near the theoretical value of 1.4 nm for full relaxation [pk93,pk90]. I also carried out structural investigations of 3C-SiC epilayers on silicon in [111] orientation [p20].

## InN

Together with PhD student Francesco Ivaldi we performed structural investigations of thick InN layers on polar and non-polar substrates and identified the density and types of defects and correlated them with the growth parameters. The non-polar layers were fully relaxed, as concluded on the basis of our lattice distortion analysis performed on HRTEM images as well as on the basis of XRD and AFM data. This result was presented at the ICNS-9 Congress in Glasgow [116] and at E-MRS 2011 in Nice [119]. The paper concerning this subject has now been accepted to the Journal of Applied Physics.

## InAlN

InAlN layers grown by MOVPE on GaN templates in the indium concentration range between 8 and 36 at% was investigated using the XANES method. The results were correlated with the local HRTEM, STEM and EDS analysis. The occurrence of a strong local gradient of composition was detected and the solubility limit of indium in AlN was determined to be near 18 at%. These results were presented at congresses [pk127, pk122, pk115] and published in [p13].

## 5.2.4 Defects

In 2011 I began a new subject of collaboration with CIMAP in France concerning the heteroepitaxy of the GaSb on GaAs and on GaP substrates. GaSb is the subject of extensive research attention due its high carrier mobility and possible applications in fast and low power consumption devices in combination with other antimonide III-V semiconductors. Collaboration in this subject originated from consulting the results obtained by Yi Wang during the preparation of his thesis. During common investigations, the atomic structure of dislocation cores of misfit dislocation was resolved using IP PAS's FEI TITAN 80-300 microscope with high-resolution STEM and Cs-corrected HRTEM techniques. These results are part of Yi Wang's thesis (the defense will take place on June 20, 2012). Up to now this collaboration has resulted in two publications [p7, accepted to APL2012].

## 5.2.4 Nanowires

## ZnTe and ZnSe

Apart from the investigation of nitride heterostructures, an important part of my scientific activity was devoted to investigating the structure of semiconductor nanowires. ZnTe-based nanowires were grown in the Laboratory of Physics and Growth of Low-Dimensional Crystals supervised by Prof. dr hab. Jacek Kossut. My activity in this domain was initiated in 2006 starting with papers [p63, pk71, p48, pk75], where together with Dr hab. Piotr Dłużewski we developed a technique of preparing nanowires for TEM observations. In these works basic data on the sizes, shapes and crystallographic orientation of ZnTe nanowires grown by MBE on [001], [110] and [111] GaAs using AuGa eutectic as a catalyst were determined. TEM

investigations showed that at the end of each nanowire the catalyst droplet is present, which demonstrated that ZnTe nanowires can grow in MBE according to the VLS (Vapor-Liquid-Solid) mechanism.

Similar results were obtained for ZnTe nanowires [p47]. The results of a more detailed structural analysis of the ZnTe nanowires were published in paper [p40]. In this paper, on the basis of series of images obtained for different tilts, I proved that the shape of the ZnTe nanowire's cross-section can change during growth from hexagonal to triangular. I also showed that the density of the stacking faults in plane [111], perpendicular to the growth direction, can vary over a wide range. I observed particular nanowires which in their upper part are defect-free. An investigation of the cross-sections of nanowire layers on a substrate carried out together with Dr hab. Piotr Dłużewski showed that the nanowires on the GaAs substrate grew mostly along the [111] B direction. An EDS chemical analysis of the interface between the ZnTe nanowire and the catalyst droplet performed together with Dr Holm Kirmse at Humboldt University in Berlin revealed the presence of Au, Ga, Zn and Te in the catalyst droplet, which is a strong confirmation of the growth of such nanowires according to the VLS mechanism. EELS and HRTEM investigations confirmed the presence of a 2-3 nm thick layer of ZnO on the surface [p38]. A detailed analysis of the HRTEM contrast of the {111} stacking faults in the ZnTe nanowires was presented at the following meetings [pk80, pk84]. Our TEM studies also showed that the catalytic ZnTe nanowires in an MBE reactor can also grow in a lateral direction exposed to the fluxes of atoms, thus causing the extension of the (111) and (0-1-1) facets [p33].

#### ZnMnTe i ZnMgTe

In all of the studied compositions for x = 0.6, the ZnMnTe nanowires crystallize in the sphalerite structure. We also observed significant structural differences in relation to non-doped ZnTe nanowires, e.g. we detected lower stacking fault densities in the {111} plane perpendicular to the axis. However, we observed NWs with SF in other {111} planes. EELS and EDS measurements showed that the distribution of manganese within NWs is homogeneous for compositions from 0 to 60% and that their surfaces are coated with a 3-nm layer of ZnO formed probably after removal of the samples from the MBE reactor [p42, p18, p26, p36, pk91].

The structure of the ZnMgTe NWs obtained by the MBE/VLS method is similar to the ZnMgTe NWS up to a concentration of x=0.3. In particular, the upper parts of the nanowires are free from defects. Above this value the nanowires still crystallize in the sphalerite structure, but their structure is not as perfect. We observe the formation of short twin segments and at the surface a thick, 10-nm layer of amorphous material is formed [pk92,p35].

#### ZnO and ZnCoO

I have been involved in research on ZnO and ZnCoO grown from vapor on a silicon substrate [p44] where I found the existence of nanowires with diameters ranging from 10 to 100 nm with a perfect, defect-free wurtzite structure.

## GaMnAs

In the case of GaMnAs nanowires grown by MBE on a GaAs substrate, TEM studies revealed the presence of multiple branches and, through the study of EDS, the presence of an increased concentration of manganese at the ends was discovered. This supported the hypothesis that the growth of such nanowires is catalyzed by the surface segregation of manganese forming of MnAs compound [p41]. In paper [p24], on the basis of TEM studies, we showed that nanowires with a manganese content below 5% grow along direction <111> and above this value in direction <110>.

#### **Core-Shell and axial nanowires**

The successful development of technology for the growth of ZnTe nanowires and diluted ZnMnMgTe nanowires using the MBE/VLS method has made it possible to grow axial and coreshell nanowires suitable for light emission. One of the problems was verifying how quickly a change of the chemical composition can be obtained in the nanowire in this kind of process.

A more extensive discussion on the example of CdTe nanowires growing at the end of the ZnTe nanowire can be found in part 4.

ZnTe-ZnO core-shell nanowires have very good absorption properties for a wide spectrum of light and their application in solar cells may be very promising. Structural HRTEM studies of ZnTe nanowires grown by MBE/VLS and covered with zinc oxide in the ALD (Atomic Layer Deposition) process show that the shells have a polycrystalline structure and form compact and sealed shells. The structure of the ZnTe core does not show any significant modifications during the ZnO coating process [p17]. It appears that a similar structure can also be obtained by oxidation of ZnTe nanowires in a controlled atmosphere. In paper [p10], by using the dark-field imaging technique, the presence of a non-oxidized ZnTe core was proven inside the ZnO shell.

The HRTEM studies and an examination of these images by analysis of the amplitude and phase in a modified GPA method revealed that the surface of the core, in contrast to the deposition of ZnO by ALD methods, was not smooth. Some of the growing crystallites penetrated into the core. However, the envelope was sealed [pk110]. It appears that the oxidation process can lead to other types of structures: nano-pipes with the wells constructed of nanocrystalline ZnO. The ZnTe core is removed and partially replaced with an alloy of Au/Te formed by the reverse movement of a gold catalyst droplet and the escape of Zn to the surface of the nanowire [pk129, pk133, pk132]

Subsequent studies performed with the use of different TEM techniques (HRTEM, EDS, SAD) in the framework of my habilitation grant [pro16] showed that it is possible to obtain CdTe nanowires with a hexagonal and polymorphic structure in the MBE process. Such nanowires form at low temperatures near 250 °C. This is below the eutectic temperature of the AuGa catalyst, which suggests that the growth takes place rather according to the VSS (vapor-solid-solid) mechanism than according to the VLS mechanism. Hexagonal CdTe nanowires nucleate on previously grown at higher temperatures cubic ZnTe NWs to form CdZnTe transition zone with the length up to 250 nm. These results were presented at the conference Microscopy of Semiconducting Materials (MSM XVII), Cambridge, U.K [126].

## 5.2.5 Other Topics

My research activity was also involved in other topics, e.g. in an investigation of the structure of laser-irradiated materials in collaboration with Dr Dorota Klingier and in the study of piezoelectric particles embedded in a PMMA polymer. In collaboration with DR hab Jarosław Stolarski from the Institute of Paleobiology PAS, the structural properties of biological composites present in coral skeletons was investigated by TEM. This entire work was the subject of congress presentations and its parts were published (see the full list of publications in Appendix 6).

This wide range of presented research may create the impression of shallowness. However, the information on the local structure is very important and cannot be obtained by other means. Without this information, it is impossible to unambiguously and correctly interpret diffraction and photoluminescence data, which are an averaged diversity existing at the nano scale. Often, even a single result sheds new light and opens the way to an appropriate interpretation. At the same time it should be remembered that TEM studies are destructive and come along with intensive and expensive labor, and therefore they cannot be used systematically for a large number of samples. Time limits allowed me to only develop some of the topics. Electron microscopy, which in recent years has gone through a technological revolution and has obtained significant hardware support in Poland, now allows investigations not only of the structure, but also of other local properties at the nanoscale level. This opens up perspectives for further research. However, it is necessary to take the effort to spread access to advanced techniques to broader groups of investigators and to open up cooperation along with the revival of the high-tech industry that has been taking place in Poland.

## 5.3 Research experience abroad

- 2003-2004 Visiting scientist; Laboratoire de Structure et Proprietes de l'Etat Solide Université des Sciences et Technologies de Lille, Francja (3 months) :
- 2000-2001 Post Doctoral Research Associate in Laboratoire CIMAP, ENSICAEN, Francja (2 years)
- 1994-1998 Phd study in Laboratories de Physiques du Solides Ecole Supérieure de Physique et de Chimie Industrielles in Paris, Francja (4 years)

## 5.4 Referee activity

Review of papers in : Journal of Alloys and Compound, Archives of Mechanics, Physica Status Solidi, Journal of Applied Physics, Solid State Phenomena.

Trans. Tech Publications.

## 5.5 Participation in research projects

Pro1	1997-2000,	Investigator, project No. PBZ 028.11/P8 from the Polish State Committee for Scientific Research, "Two-, one- and zero-dimensional semiconductor
		heterostructures of group II tellurides and diluted magnetic semiconductors within a perspective of optoelectronics applications", Institute of Physics
Pro2	1999-2001.	main investigator 7T07A00416
	,	"Tensorial measures of structural defects in prediction of the stress and
		lattice distortion distribution in epitaxial layers" Institute of Fundamental
		technological Research PAS.
Pro3	1998-2002,	investigator, P03B 103 14,
		"Nanostructures in the light of the X-ray and electron microscopy
		research"struktury w świetle badań rentgenowskich i elektrono
		mikroskopowych", Institute of Physics PAS,
Pro4	2000-2004,	investigator, G5RT-CT-2000-05001,
		European scientific network "Metal oxide multilayers obtained by cost-
		effective new CVD technologies for magnetoelectronic microsystems and
		University of Barcelona Spain
Pro5	2000-2004	investigator, ICA1-CT-2000-70018.
		CELDIS "Physics and Fabrication of Low Dimensional Structures for
		Technologies of Future Generations",
Pro6	2003-2004	principal investigator of PAN-CNRS. Project nr 14476:
		"Analysis of local atomic structure of heterostructures", Institute of
		Physics PAS.
Pro7	2003-2005	investigator, G1MA-CI-2002-4017 (CEPHEUS)
		"Centre of Photon, Electron and Ion Advanced Methods for Natural
D0	2004 2007	Science", IPPAS
Proð	2004-2007	main investigator, 4 T07A 010 26,
		the formation of quantum data in a nituida land the formation of quantum data in a nituida land
		technological Research, DAS
Pro9	2006-2008	investigator N507.020.21/0725
	2000 2000	"Catalytic growth technology by molecular beam enitaxy of
		semiconductor nanowires AIIBVI for the needs of future electronics"
		Institute of Physics PAS.
Pro10	2007-2010	investigator, N515 015 32/099.7
		"ZnO nanowires :technology and properties", Institute of Physics PAS.
Pro11	2007-2010	investigator, N202 052 32/1189,

	" MnSb based magnetic composites for future nanoelectronics", Institute of Physics PAS
Pro12 2007-20	010 investigator N N202 0634 33,
	"Electrical control of spin state of the CdTe quantum dots doped with
	manganese", Institute of Physics PAS
Pro13 2007-2	013 investigator, POIG.01.01.02-00-008/08,
	"Semiconductor quantum structures for applications in biology and
	medicine", Institute of Physics PAS
Pro14 2008-20	012 principal investigator in IP-PAS of European project RAINBOW,
	213238-2, FP7-PEOPLE-2007-1-1-ITN. The People Programme, Initial
	Training Networks,"High quality material and intrinsic properties of InN
	and indium rich nitride alloys", coordinator CIMAP, Universytet Caen,
	France
Pro15 2009-2	2011 main investigator, POIG.02.01.00-14-032/08, "Analytical, high
	resolution transmision elektron microscope for nanoscience,
	nanotechnology and spintronics" Institute of Physics PAS.
Pro16 200-20	0011 principal investigator, SpubM decyzja NR 1242/7.PR UE/2010/7 z dnia 5
	lutego 2010: "Wysoka jakość i właściwości materiałów opartych na InN i
	innych półprzewodnikach azotkowych bogatych w ind", Institute of
	Physics PAS.
Pro16-2010-20	principal investigator, N N202 204438
	"New methods for the quantitative transmission electron microscopy in
	physics and technology of semiconductor nanostructures", Institute of
	Physics PAS.

## 5.6 Other achievements

## Didactic

- Prowaiding in the yers 2006-2012 lecture: Transmisyjna Mikroskopia Elektronów" in the frame of a series of lectures: Fizyka, technologia oraz modelowanie wzrostu kryształów, ICM 90 minut.
- 2. "QHRTEM as the practical method for semiconductor heterostructures characterization", 2h of lecture and 10h of exercises during school: "Escuela TEM UCA 2004 "Microscopia Electronica con Resolucion Atomica", Universidad de Cadiz, Spain
- Summer School on Stress/Strain Determination by TEM Methods, Wisla 14-16 September 2006. 4h of exercises of practical application of computer methods for analysis of HRTEM images.

- 4. 14h of exercises in the field of quantitative analysis of the HRTEM images of an international workshop organized within the center Cepheus, (effect of these activities is to promote the image analysis techniques HRTEM Cadiz University, Tesalonick University).
- 5. Supervision of research and consultation results PhD student Dipl. eng. Francesco Ivaldi (defense scheduled for September 2012)
- Consulting of the a part of the results of Phd student Yi Wang from Caen University, (defense scheduled for 20 Jun 2012)
- 7. Consulting of the a part of the results of Phd student Teresy Ben, Cadiz University (defended in 2006).

## Work in organizations

- Participation in the scientific and organizing committee of the conference entitled: Experimental and Computing Methods in High Resolution Diffraction Applied for Structure Characterization of Modern Materials, HREDAMM, Zakopane, Poland, June 13-17, 2004 <u>http://info.ifpan.edu.pl/cepheus/HREDAMM2004/index.htm</u>.
- Co-organizer of the workshop entitled: International Mini-Workshop on Practical Aspects of Quantitative Analysis of HRTEM Images, April 19-23, 2004 Warsaw, Poland, <u>http://info.ifpan.edu.pl/cepheus/mw/mwprog.html</u> in the framework of the Center of Excellence CEPHEUS, 5 days, 30 participations from 3 countries, 10h of exercises.
- Chairman of the organizing committee for the workshop entitled: Workshop on Advanced Method for Interpretation of TEM, X-Ray and SIMS Measurements in Nano and Atomic Scale, June 3, 2005, Warsaw, Poland.

A three-day workshop, 18 lectures, 5h of exercises, 10 invited speakers from abroad due to the support of the Center of Excellence CEPHEUS, 72 participants from 8 countries, <u>http://info.ifpan.edu.pl/cepheus/Workshop/WORKSHOP2005.htm</u>.

## **Popularizing science**

- In the years 1999–2000 – Lessons for groups of students from high schools and universities on "Transmission Electron Microscopy".

- In the years 2003–2006 – Festival of Science, a presentation of the methodology of transmission electron microscopy.

- In the years 2006-2010 - Evaluating the work of the Young Physicists Tournament.

- 2011 Preparation and evaluation of qualification tasks in workshops for talented young people organized by the National Children's Fund.

- Research workshop on the physics of magnetism, superconductivity, semiconductors and biophysics at the Institute of Physics PAS, January 23–28, 2011 for talented youth organized by the National Children's Fund, overlooked two trainees (Kacper Łanda, Michał Glapa) working on the subject of: Fast electrons reveal the secrets of crystals or microscopy with atomic resolution.

- Participation in a popular science film: "The value of the electron" in the framework of project [pro15].

- Participation in a popular science film: "Micro Nano Piko" in the framework of project [pro15].

## International cooperation

- 1. 2000-2012- directeurs de recherche P. Ruterana, Le Laboratoire l'ENSICAEN-CNRS Uiversité de Caen, France,
- 2. 2008-2012 Projekt PAN-CNRS, Projekt RAINBOW international consortium of 13 partners
- 3. 2004-2008- professor S. Molina, dr P. Galindo, Department of Materials Science and Metallurgical Engineering and Inorganic Chemistry, University of Cadiz; Hiszpania
- 3. prof W. Neumann, Dr. rer. nat. H. Kirmse, Uniwersytet Humboldta , Berlin, Germany
- 4. 2008-2001 M. Zielinski, NOVASiC, Savoie Technolac, Le Bourget du Lac, France
- 5. 1993-2008 dr J-Y. Laval, Laboratoire de Physique du Solide, l'Ecole Supérieure de Physique et de Chimie Industrielles de la Ville de Paris, France
- 7. 1996-2006 dr A. Lefebre, Y. Androussi, Université des Sciences et Technologies de Lille, France
- dr M. Hytch, Centre d'Elaboration de Matériaux et d'Etudes Structurales, CNRS w Touluzie France
- 10. dr Ana Sanchez, Universytet Worwick, England

#### Participation in laboratory development

In 1998 – participation in a project whose aim was to purchase a high-resolution electron microscope with field emission for the LPS-ESPCI in Paris. I was involved in the preparation of the technical design and testing of microscopes and accessories from manufacturers.

In 2008 the transmission microscope at IP PAS that had been operating continuously for 20 years failed to meet expectations for the quality of results due to limited resolution and the lack of analytical capacity. In the years 2008–2011 I became involved in a project whose aim was to purchase and install a new transmission electron microscope. I consider my contribution to this project, coordinated by Dr hab. Piotr Dłużewski, to have been significant. Together with Dr hab. Piotr Dłużewski we developed the concept of the technical and scientific project. I participated in preparing the application for the project, creating the specifications of the tender, formulating the concept of adapting the building's basement, supervising the building adaptation process and, most importantly, supervising the installation of the transmission microscope as well as the scanning electron microscope (with the possibility of cutting TEM lamella with the use of a focused ion beam (FIB)) http://awtem.ifpan.edu.pl/Kronika/index.html).

After successful start-up of the apparatus, in the years 2010–2012 I was involved in the process of obtaining accreditation for the Laboratory of Electron Microscopy. This process was successfully completed by its obtaining a certificate of completion from the PCA. I currently have the position of technical coordinator in this laboratory.

## 5.7 List of presentations at international and national conferences and thematic workshops

- "Cartographie á l'échelle atomique des contraintes dans des boîtes quantiques GaAs/As(1x)Inx fortement désadaptées", S.Kret, C.Delamarre, A.Dubon, J-Y.Laval, Première Rencontre Franco-Caribéenne Pointe-â-Pître, Guadeloupe, France, 26-29 Mai 1997
- 2. "Analysis of strain in the {1120} prismatic fault in GaN using digital processing of highresolution transmission electron microscopy images", **S.Kret**, P.Ruterana, G.Nouet, Int. Conf. on Extended Defects in Semiconductors, Brighton, UK, 18-22.07.2000,
- 3. "Investigation of threading dislocation atomic configurations in GaN by HRTEM, geometrical phase analysis and atomistic modelling", **S.Kret**, J.Chen, P.Ruterana, G.Nouet, Microscopy of Semiconductive Materials, Oxford, 25-29 March, 2001
- 4. "Sample preparation for TEM", **S.Kret**, MULTIMETOX TEM'2002, International Workshop on Microstructural Characterisation of Oxide Films and Multilayers by TEM and HREM, Warsaw, Poland, 26-27 Sept. 2002,
- "Quantitative transmission electron microscopy investigation of localized stress in heterostructures", S.Kret, P.Dłużewski, G.Maciejewski, G.Jurczak, P.Ruterana, J.Chen, P.Dłużewski, E.Janik, E-MRS 2003 Fall Meeting, Warsaw, Poland, 15-19 Sept., 2003,
- "Quantitative electron microscopy of the semiconductors nanostructures, Part I/PartII, Extraction of the quantitative data from HRTEM images: on-line demonstration of image processing", S.Kret, Intergranular and Interphase Boundaries, Experimental Techniques of Investigation and Computer Simulation Methods Workshop, Politechnika Warszawska, 01-04.09.2004, (INVITED)
- 7. "Introduction to Geometric Phase Methods of strain measurement", **S.Kret**, International Mini-Workshop on Practical Aspects of Quantitative Analysis of HRTEM Images, Warsaw, 19-23.04.2004,
- 8. "Workshop targets-QHRTEM overview", **S.Kret**, Int. Mini-Workshop on Practical Aspects of Quantitative Analysis of HRTEM Images, Warszawa, Polska, 2004
- "Application of the FEM in quentitative high resolution transmission electron microscopy": Part I, S.Kret, Summer School on Stress/Strain Determination by TEM Methods, on XX Conf. on Applied Crystallography, 14-16.09.2006, Wisła, Poland (INVITED)
- "Application of the FEM in quentitative high resolution transmission electron microscopy", S.Kret, Summer School on Stress/Strain Determination by TEM Methods, on XX Conf. on Applied Crystallography, 14-16.09.2006, Wisła, Poland, (INVITED)
- "Quantitative high resolution transmission electron microscopy", S.Kret, Summer School on Stress/Strain Determination by TEM Methods, on XX Conf. on Applied Crystallography, 14-16.09.2006, Wisła (INVITED)
- 12. "Analysis of diffraction contrast transmission electron miccroscopy images of strained nanostructures", **S.Kret**, NANOTEM SCHOOL, Cadiz, Hiszpania, 2008, **(INVITED)**
- 13. "Quantitative TEM of InN and XRD investigations", S.Kret, Kick-off meeting projektu RAINBOW, Crepon, Francja, 2008
- "The asymmetry of planar defect density in 3C-SiC grown on disoriented silicon substrates", S.Kret, A.Szczepańska, M.Zielinski, T.Chassagne, M.Portail, 7-th Polish-Japanese Joint Seminar on Micro and Nano Analysis, Warszawa, Polska, 2008

- 15. "Nanoscale disorder in heterostructures based on nitride semiconductor determination by STEM, HRTEM and EFTEM", **S.Kret**, A.Letrouit, F.Ivaldi, A.Szczepańska, B.Kurowska, XIVth Int. Conf. on Electron Microscopy, Wisła, Polska, 2011, **(INVITED)**
- "Spontaneous structural transformation of MOVPE InAlN epilayers on GaN templates", S.Kret, F.Ivaldi, A.Letrouit, A.Szczepańska, J.Carlin, N.Kaufmann, N.Grandjean, E-MRS 2011 Spring Meeting, Nicea, Francja, 2011
- "TEM and XANES study of MOVPE grown InAlN layers with different indium content", S.Kret, A.Wolska, M.Klepka, F.Ivaldi, J.Carlin, N.Kaufmann, N.Grandjean., MSM XVII Microscopy of Semiconducting Materials 2011, Cambridge, W.Brytania, 2011
- "TEM investigation of high indium containing InGaN precipitations grown by MOVPE", S.Kret, F.Ivaldi, A.Kadir, C.Meissner, T.Schwaner, M.Pristovsek, B.Kurowska, M.Kneissl, M.Klepka, 9th Int. Conf. On Nitride Semiconductors, Glasgow, W.Brytania, 2011

## 6.6 List of delivered seminars

- Se1 "Elektronomikroskopowe badanie niejednorodności strukturalnych w studniach i kropkach kwantowych", **S.Kret**, Seminarium fizyki materiałów, Wydział Fizyki UW, 14.12.1999
- Se2 "Elektronomikroskopowe pomiary lokalnych dystorsji sieci w heterostrukturach półprzewodnikowych", S.Kret, seminarium z fizyki ciała stałego, Wydział Fizyki UW, 24.03.2000
- Se3 "Zastosowanie wysokorozdzielczej transmisyjnej mikroskopii elektronowej do pomiaru lokalnych dystorsji sieci w półprzewodnikowych warstwach epitaksjalnych", S.Kret, Seminarium Ośrodka Mechaniki, Instytut Podstawowych Problemów Techniki PAN, 10.03.2000,
- Se4 "Pomiary lokalnych dystorsji sieci w zdefektowanych warstwach GaN", **S.Kret**, seminarium Zespołu Mechaniki Mikro i Nano Struktury Kryształów, Instytut Podstawowych Problemów Techniki PAN, 03.12.2000,
- Se5 "Ilościowa Mikroskopia elektronowa w badaniach nanostruktur", **S.Kret**, Seminarium Rentgenowskie, IF PAN, 5.02.2003
- Se6 "Image processing and finite element modeling in quantitative interpretation of HRTEM images", **S.Kret**, seminarium laboratorium Laboratorie de Structure et Propriétés de l'Etat Solide, Université des Sciences et Technologies de Lille, 7 listopad 2003.
- Se7 "Charakteryzacja kropek kwantowych InGaAs i CdZnTe metodami ilościowej mikroskopii elektronowej", S.Kret, Seminarium Badań Strukturalnych, Instytut Fizyki Doświadczalnej, Wydział Fizyki UW, 17.11.2004
- Se8 "Application of the strain measurement from HREM to chemical composition and piezoelectric field determination in InGaN and CdZnTe nanostructures", **S.Kret**, seminarium, Uniwersytet Humboldta, Berlin, maj 2006
- Se9 "Quantitative TEM of InN and XRD investigations", **S.Kret**, Kick-off meeting projektu RAINBOW, Crépon, France, 3- 5 November 2008
- Se10 "Mapowanie koncentracji indu w strukturach kwantowych InGaN/GaN metodami transmisyjnej mikroskopii elektronowej", S.Kret, Seminarium Rentgenowskie w IF PAN, 2.06. 2009
- Sel1 "Parametry techniczne i możliwości badawcze mikroskopu FEI Titan CUBED 80-300", S.Kret, Seminarium AWTEM, "Zaproszenie do współpracy" w ramach projektu

"Analityczny, wysokorozdzielczy, transmisyjny mikroskop elektronowy dla nanonauki, nanotechnologii i spintroniki", POIG-6, IF PAN, Warszawa, 17.06.2010 r

- Se12 "Pracownia Mikroskopii Elektronowej IF PAN, Parametry techniczne i możliwości badawcze mikroskopu FEI Titan CUBED 80-300 oraz HELIOS 600, S.Kret, uroczystość otwarcia PME w ramach projektu POIG6, IF PAN, Warszawa, 10.12.2010
- Se13 "TEM and XRD of In reach nitride alloys", S.Kret, Rainbow Mid-Term Meeting, Madrid, 30 August 2010,
- Se14 "Transmisyjna mikroskopia skaningowa STEM", S.Kret, Seminarium AWTEM, Seminarium promocyjne projektu AWTEM, "Możliwości pomiarowe mikroskopu elektronowego Titan-Cubed 80-300", 30.03. 2011
- Se15 "Skaningowa transmisyjna mikroskopia elektronowa", S.Kret, "Od instalacji do eksploatacji", Seminarium promocyjne projektu AWTEM, S.Kret, Seminarium AWTEM - "Analityczny, wysokorozdzielczy, transmisyjny mikroskop elektronowy dla nanonauki, nanotechnologii i spintroniki", Instytut Fizyki PAN, 26.09.2011

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