



# Oponentní posudek disertační práce

Ústav: Středoevropský technologický institut VUT Akademický rok: **2020/2021**

Student (ka): **Ing. Petr Vacek**

Doktorský studijní program: **Pokročilé materiály a nanovědy**

Studijní odbor: **Pokročilé nanotechnologie a mikrotechnologie**

Vedoucí disertační práce: **doc. Ing. Roman Gröger, Ph.D. et Ph.D.**

Oponent disertační práce: **Priv.- Doz. Dr. David Holec**

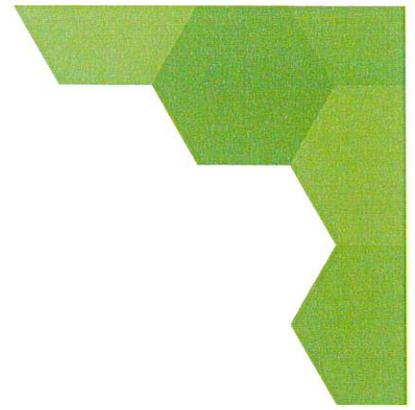
Název pojednání práce: **Rozsáhlé defekty v nitridech Ga a Al**

## Aktuálnost tématu disertační práce:

About 20 years ago, III-nitrides based on Ga, Al and In had revolutionised the field of optoelectronics. Mastering their synthesis allowed the Blu-ray technology and boom of solid-state lighting by allowing for white LEDs with tunable spectrum, to name a few examples. However, their performance critically depends on the material quality in terms of type and density of contained defects which, in turn, is determined by the deposition procedure. Another essential factor in this respect is the choice of the substrate. Initially, the technology was developed using sapphire. However, it is desirable to establish it also for Si substrates since they are cheaper, larger and overall, the Si technology is probably the most mature one in the semiconductor field. Nonetheless, growth on Si has not been yet fully optimised. Therefore, the main focus of this thesis, experimental characterisation of the type and role of extended defects (dislocations and stacking faults) of AlN and GaN grown on Si(111) and SiC/Si(100) substrates, is of great relevance both scientifically as well as technologically.

## Splnění stanovených cílů:

Chapter 3 lists six aims of the thesis, grouped into two groups: defects in wurtzite III-N and defects in zincblende III-N. All those aims are discussed and answered in the present thesis, leading to new insights and understandings. Perhaps the goals could have been formulated slightly more specific, e.g. instead of zincblende III-N, use zb-GaN (as other zb-III-N have not been attempted in the present thesis), but this is only a minor issue. Moreover, those details are provided right above the list of the goals.



### **Postup řešení problému a výsledky disertace:**

The presented work comprises experimental investigations which combine several state-of-the-art characterisation techniques. These include atomic force microscopy (AFM), scanning and transmission electron microscopy (SEM and TEM), X-ray diffraction and a relatively uncommon but well-suited correlative probe and electron microscopy. The presented results suggest that the candidate has mastered those techniques.

Surface features and their in-plane distribution are characterised using AFM. For example, it was concluded that the initially isolated island of w-AlN grown on Si(111) fully coalesce between 10 and 30 nm of film thickness. They also revealed surface depressions of different sizes assigned to so-called V-defects and terminations of dislocations. High-resolution TEM (HRTEM) micrographs revealed an amorphous interlayer between the Si substrate and the AlN layer. Plan view bright-field images proved the existence of threading dislocations (TDs), whose density significantly reduces in the GaN layer grown on top of AlN/Si(111). It has been concluded that the most probable mechanism for their formation is bending of misfit dislocations at the substrate/film interface upwards into the film-growth direction and reactions of partials bounding stacking faults. The electrical activity of the defects was investigated by electron beam induced current (EBIC). Those measurements suggest charge carrier losses at those defects.

The second part of the thesis characterises zb-GaN grown on SiC/Si(100) substrates with miscut. The growth starts with an island-like morphology, with the island elongated in the direction of miscut. Annealing experiments lead to larger surface features and consequently somewhat reduced surface coverage. The island elongation was also linked with the different facet angle (front vs Back) due to the substrate miscut. The last part deals with a detailed microscopy investigation on stacking faults (SFs) structure in zb-GaN. Many beautiful HRTEM images were presented, and the reviewer wishes to congratulate Petr on them and their compelling analysis. Based on TEM images, a model for SF density reduction is presented.

### **Význam pro praxi nebo rozvoj vědního oboru:**

The simple fact that the project was done in collaboration with a company (ON Semiconductor) suggests that the topic is industrially and technologically relevant. The investigated samples were grown by the company partner, and hence the detailed characterisation performed in this thesis can be directly applied for optimisation of the growth processes. Dislocations were identified as non-radiative recombination regions before. However, here this fact is also proved for dislocation in AlN and GaN grown on Si, hence exhibiting presumably very different stress state than in the case of sapphire substrates. The comprehensive analysis and characterisation of stacking faults in GaN are unique and, to the reviewer's best knowledge, has not been reported in the literature before.

### Formální úprava disertační práce a její jazyková úroveň:

The thesis has 91 pages, including preface and references, and the content is divided into six chapters. All figures and table contain captions with sufficient explanation, and the presented figures are in high enough resolution. The style of the figures and the overall formatting is consistent throughout the thesis and makes an excellent impression. The thesis is written in English with clearly communicating the intended message. The reviewer has not spotted any grammatical errors. The only minor typos found are "Blue-ray technology" (page 15) and the name of the host institution in the UK, which, according to the reviewer's knowledge, is "Department of Materials Science & Metallurgy" (3x on pages 29 and 30).

### Zda dizertační práce splňuje podmínky uvedené v § 47 odst. 4 zákona:

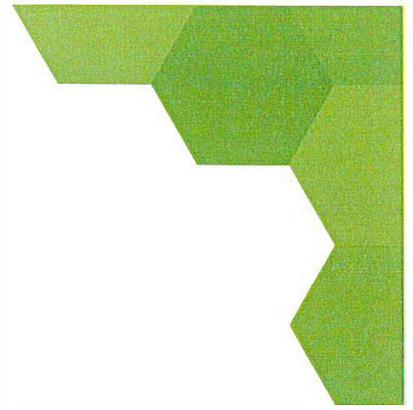
(4) Studium se řádně ukončuje státní doktorskou zkouškou a obhajobou disertační práce, kterými se prokazuje schopnost a připravenost k samostatné činnosti v oblasti výzkumu nebo vývoje nebo k samostatné teoretické a tvůrčí umělecké činnosti. Disertační práce musí obsahovat původní a uveřejněné výsledky nebo výsledky přijaté k uveřejnění.<sup>1)</sup>

To the reviewer's best judgement, the thesis of Petr Vacek title "Extended defects in Ga and Al nitrides" fulfils the requirements mentioned above and should therefore be admitted for defence.

### Připomínky a dotazy:

1. Why was the description of EBIC (Section 2.5) not included among other characterization methods (Chapter 3)? Also, the description of "tailing" in Fig. 2.12 is not completely clear to a non-specialist. Please provide a schematic picture of how does it appear? Which settings (scanning speed) eliminate those artefacts?
2. It is unclear how the epitaxial relationship from the substrate can be maintained in the AlN layer when an amorphous layer forms at the film-substrate interface (page 32). Please elaborate.
3. It is claimed that the thickness of the amorphous layer increased later on due to the diffusion of Si and N (page 35). What about Al? Have the diffusion coefficients been estimated? Is Si in AlN expected from thermodynamics?
4. What is the Burgers vector of the misfit dislocation shown in Fig. 5.7? The atomic arrangement and shown schematics suggest only an edge component along the  $\langle 0001 \rangle$  direction, which does not relieve any misfit strain.
5. On page 41, the author reports statistics of different dislocation types. The fractions are very accurate – up to 3 decimal place (1 when shown in percents). How many dislocations/images were overall included in this statistical evaluation? How reproducible are those numbers from various deposition batches using identical deposition conditions?
6. The molecular statics simulation in Fig. 5.12 is excellent. Presumably, this is the author's own work since there is no reference. Please provide some more details about the modelling. Were

<sup>1)</sup> § 10 zákona č. 35/1965 Sb., o dílech literárních, vědeckých a uměleckých (autorský zákon).



- different geometries and or more sizes attempted? What about temperature effects? Cannot that enhance the force for creating Tds?
7. Since the c-type dislocations do not relieve misfit strain, they are not obtained by the bending mechanism. What is their origin?
  8. How is the EBIC contrast  $C_{max}$  related to the defect density in Table 1 (page 52)?
  9. An own Monte Carlo simulation is mentioned on page 54, but without any reference, details are showing any results. More details should be provided.
  10. The second part of the results deals with zincblende (zb) GaN. A motivation in terms of the advantages of the metastable zb-GaN over its stable wurtzite polymorph is missing.
  11. Figure 5.25 misses the dashed reference line for fully relaxed GaN, as mentioned in the figure caption.
  12. Please elaborate on the differences between AFM and TEM reported in Table 2. The difference between the angle for the front and back facets is twice as large in TEM than in AFM! Why?
  13. The stacking fault reduction model is very similar to models used for predicting dislocation density reduction. The reaction table (Table 4) refers to the 3D reality, whereas the model qualitative was 2D (page 75). How was the information from Table 4 transferred into the 2D model?
  14. The comparison between theory and experiment in Fig. 5.39 is nice. Most of the predicted SF reduction is predicted within the first few dozens of nanometers. However, in this region (Stage I), only a single experimental point is shown, which makes the comparison somewhat arbitrary. How was the initial SF density for the model chosen? It is a critical parameter for predictions!

#### Celkové zhodnocení disertační práce:

The overall impression of the work is very positive—it present novel and exciting results in a high scientific and formal quality. The thesis provides explanations for the studied phenomena, which are well illustrated by the numerous figures. Equally important is to state that the results are critically assessed, thus avoiding speculations and overinterpretation. The reviewer read the thesis with great interest, and he has undoubtedly learned new facts about the fascinating III-N materials. The reviewer congratulates Petr on this thesis which fulfils all criteria put on PhD thesis and is therefore very happy to recommend it for approval.

Disertační práci Ing. Petra Vacka doporučuji k obhajobě pro udělení akademického titulu "doktor" (Ph.D.).

V Leobenu, dne 22. června 2021



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Priv.- Doz. Dr. David Holec

