Projekt zum Fortgeschrittenen-Praktikum - Rasterkraftmikroskopie

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0 Expose

The III-V semiconductor system based on GaSb is commonly used for optical semiconductor devices with wavelengths beyond 2.3 µm [1]. In Würzburg especially the interband cascade lasers, which are grown by MBE on GaSb substrate, made significant progress during the last years [2]. In order to grow devices with high performance it is inevitable to use high quality substrates with a minimum of defects. Despite the use of 'epiready' substrates the wafers suffer from native oxide like Ga_2O_3 and Sb_2O_3 [3]. The growth of devices on top of this oxide would lead to non-monocrystal layers. To remove this oxide a commonly used technique in Würzburg is to heat the substrate to about 580° for a short time. At this temperature the most of the oxide desorbes from the surface but leaving holes in the surface with about 10 nm in size [4]. Hereupon a 200 nm GaSb buffer layer is grown at 485° to flatten the surface.
This method has been established during the last years although it is not clear whether a different technique would lead to smoother surfaces. Therefore one of the goals of this experiment is to determine a method of reducing the roughness on the wafer we want to grow on optical structures. This is important on the behalf of the optical quality the device can operate at.

From an intuitive point of view it is clear what roughness is. However, quantifying roughness in a mathematical way is not trivial. Common definitions use the standard deviation of the surface's mean height as $S_a = \frac{1}{A} \iint\limits_A |z(x,y)| \mathrm{d}x\mathrm{d}y$ and are suitable in many applications. Problems arise when applying this definition to non-flat surfaces. A soup bowl which appears flat and smooth when we are very close to the surface, shows a curvature when we look at the object as a whole. This example shows, that roughness is not independent of the scale. In our scope roughness will be a way of measuring the quality of a surface with respect to certain properties of the material. We think that in general the concept of a surface energy is a better approach to address the optical properties arising from surface irregularities. The more the size of the surface differs from a perfect flat surface the rougher the surface is. We want to model the surface as polygons connecting the mean heights of a discrete lattice. The lattice size represents the scaling parameter mentioned above. Here we make the arbitrary choice of the AFM's resolution which will be the scale on which we measure the roughness of the samples.

The Atomic Force Microscope (AFM) is the perfect instrument to characterize this roughness of the wafers as it determines the height of the surface very precisely. The expected differences in height on the surface is about 10 nm which is within the resolution of the AFM. As the AFM doesn't work in situ we have to produce and investigate the surface at each step of the growth process to understand the mechanisms of oxide desorbtion and flattening of the surface. We are going to characterize the single steps of the standard process which are: an untreated GaSb wafer, the wafer after the oxide desorbtion and after the growth of 200 nm GaSb buffer. To vary this process we want to test two aspects: first the increase of the GaSb buffer's growth temperature up to 500° and 515° and second the growth of a 30 nm GaSb/AlAsSb superlattice directly after oxide desorbtion. Recent research showed that the growth of

Abb. 1: At the sample's surface after several micrometer growth small pyramidal defects are visible. The image was taken by an optical microscope at a magnification of 50.

AlAsSb shutting down the step-flow growth mode. This step-flow growth mode is dominant during the growth of GaSb layers and is not very successful in flattening bigger defects like defects in pyramidal shape [4]. The growth of a superlattice instead of a bulk layer is nevertheless necessary to maintain the electrical conductivity of the sample. It would be helpful to understand how these defects can be removed from the surface and how the process can be improved. If the smoothing is not successful theses pyramidal defects tend to grow bigger as the growth progresses. After the growth of structures with a thickness of several microns these defects can even be observed by a optical microscope as shown in Abb. 1.

After sample production exposure to air can not be avoided. To reduce surface corrosion the samples will be produced tight before the experiment and stored in a nitrogen-flooded cabinet.

1 Basics

The relevance of fabrication and therefore also the analysis of structure in the sub-micrometer and even nanometer scale has increased steadily in the past decades. In contrast to other scanning imaging techniques as the STM or SEM, the atomic force microscope (AFM) is capable to deliver up to atomic resolution without the need of a vacuum or special treatment of the probe prior to analysis. This allows us to examine a broader variety of samples under easily achievable conditions.

Funktionsweise AFM
Relevanz für die Aufnahme der GaSb Proben

2 Data Aquisition

In order to acquire the topology of the samples surface the computer records the scanner's z-position in combination with the in plane x- and y-position. These data can be exported from the control software in a special (.ezd) data format. We convert these data back into a list of x, y and z-coordinates using the *WSxM* software, which is freely available on ??.

While scanning in static force mode is necessary to maintain the force by moving the scanner's z-position. This is done by a feedback loop, which compares the a stated force constant to the force calculated from the signal of the photodiode. The difference is called the error signal and is the basis for the PI-controller. This controller includes to variables, the P- and I-values, which can be adjusted at the control software. While recording the data we can adjust the P- and I-values to optimize the quality of the pictures and avoid image artifacts. These values refer to the proportional and integral gain of the z-controllers feedback loop respectively. The proportional gain provides a signal which is proportional to the error signal. By increasing this value one should observe a smaller static error signal and a faster adaption to the current height position. On the other hand, if this value is set to high, one should observe a overshoots while scanning steps. Even higher P-values lead to more noise as the scanner is reacting oversensitive to little changes in height. The I-value however provides a signal which is proportional to the temporal integral of the error signal. The adaption of this value may have similar effects as adaption the P-value. Nevertheless the signal from the integral controller is lee sensitive to noise.

In the beginning of the experiment we scanned a calibration grid while varying the P- and I-parameter to see the effects on the pictures quality. The calibration grid consists of a grid with two different height levels. By scanning the grid in one direction one should observe steps in the up and down direction. In the following pictures every scan is conducted from the left the right side. Firstly we started with a low I-value and observed the z-coordinate adepting very slowly to the actual height of the grid as is shown in 2.

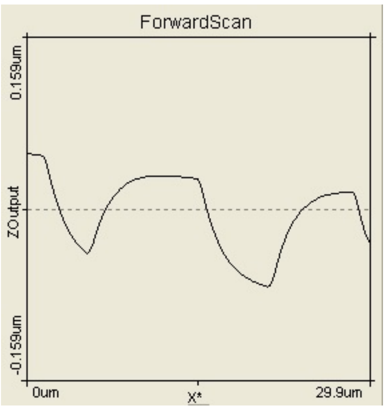

Abb. 2: Height profile recorded with the I-gain value set to 5. P = 9. Scanning direction is from left to right.

By increasing the I-value the height is adapting much more quickly but overshoots at the step's edge as well as increasing the amount of noise during the plateaus. The height profile is plotted in 3.

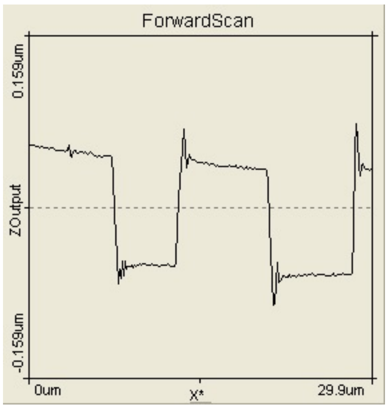

Abb. 3: Height profile recordes with the I-gain value set to 11. P = 9. Scanning direction is from left to right.

In the next step the influence of the P-value was observed. With the I-value set to 9 the p-value was varied from 5 to 15. Interestingly an decrease of the P-value to 5 doesn't change much but increasing the overshoots caused by the I-value, which is set little higher than the optimum value. Simultaneously the overshoots decrease when increasing the P-value. This however has a negative influence on the noise as can be seen in the comparison of 4 and 5.

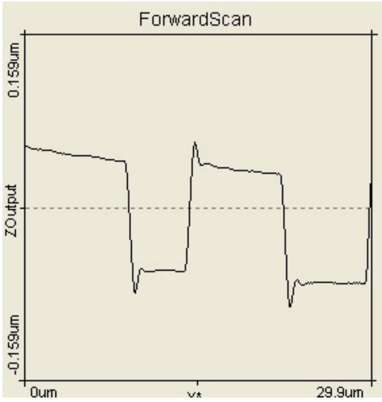

Abb. 4: Height profile recordes with the P-gain value set to 5. I = 9. Scanning direction is from left to right.

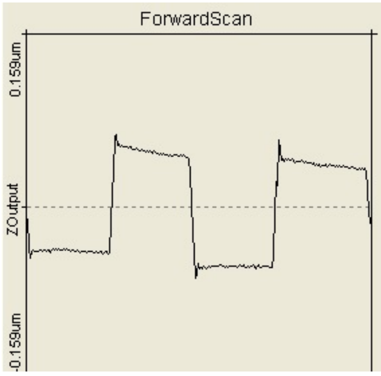

Abb. 5: Height profile recordes with the P-gain value set to 15. I = 9. Scanning direction is from left to right.

As a trade-off between fast and accurate height adaption and a minimum of noise we choose I = 9 and I = 7. We use these parameters in all following measurements, if not stated differently.

Next we want to investigate what influence the speed of scanning has on the image recording. We measure the calibration grid with 0.7 s and 3 s per line. During this period 256 data points are recorded along the scanning axis. For the slow scan speed we observe in 6 the clear step of the calibration grit without any noticeable overshoot. For the faster scanning speed we can see in

7 distinct overshoots at the beginning of each step.

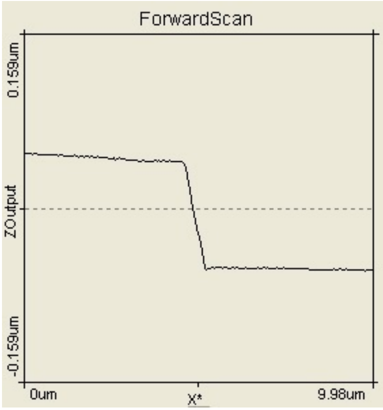

Abb. 6: Height profile recordes with a scanning sped of 3 s per line. P = 9, I = 9, scanning direction is from left to right.

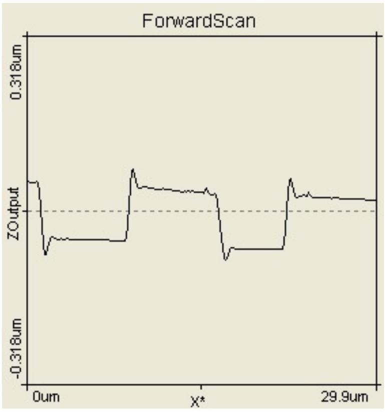

Abb. 7: Height profile recordes with a scanning sped of 0.7 s per line. P = 9, I = 9, scanning direction is from left to right.

Although we observe artifacts while scanning the calibration grit with 0.7 s per line it is possible to measure structures without steep gradient with a higher scanning speed without observing overshoots.

- Rauschen- SNR Arten

- Methoden und Probleme der Bildbearbeitung

3 Sample Analysis

- Features

- Rauigkeit der Oberfläche

- Aussagen über Wachstumsmethoden

Literatur

[1] Shamsul Arafin (2012): Electrically-Pumped GaSb-Based Vertical-Cavity Surface-Emitting Lasers. München.

[2] Weih, Robert; Kamp, Martin; Höfling, Sven (2013): Interband cascade lasers with room temperature threshold current densities below 100 A/cm2. In: Appl. Phys. Lett. 102 (23), S. 231123. DOI: 10.1063/1.4811133.

[3] C.J. Vineis; C.A. Wang; K.F. Jensen (2001): Insitu reflectance monitoring of GaSb substrate oxide desorption 2001.

[4] Murray, Lee M.; Yildirim, Asli; Provence, Sydney R.; Norton, Dennis T.; Boggess, Thomas F.; Prineas, John P. (2013): Causes and elimination of pyramidal defects in GaSb-based epitaxial layers. In: J. Vac. Sci. Technol. B 31 (3), S. 03C108. DOI: 10.1116/1.4792515.