

Projekt zum Fortgeschrittenen-Praktikum

- Atomic Force Microscopy

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1 Expose

The III-V semiconductor system based on GaSb is commonly used for optical semiconductor devices with wavelengths beyond $2.3\text{ }\mu\text{m}$ [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 'epi-ready' 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 desorbs 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 $S_a = \frac{1}{A} \iint_A |z(x, y)| dx dy$ 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 desorption 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 desorption 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 desorption. Recent research showed that the growth of



Figure 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 these 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 an optical microscope as shown in figure 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.

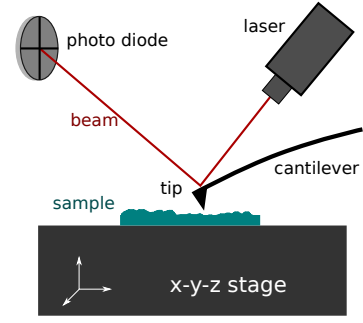


Figure 2: Simplified illustration of an atomic force microscope

2 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.

2.1 Work principle of the AFM

The principle of the atomic force microscope are the forces that act between two pieces of matter when brought close to each other. More precisely we bring a sharp tip into a distance of a few nano meters to a surface we want to examine. The main contribution are Van-der-Waals forces which arise from polarisation fluctuations in the material. In figure 2 one can see the main parts required to run the AFM. The sample is mounted on a xyz-stage consisting of different piezo elements. This enables us to move the sample in xy-direction for the scanning of the probe, as well as in the z direction for height compensation. A reflecting cantilever is positioned above the sample. A laser beam will reflect on the cantilever and hits a segmented photodiode. If the cantilever moves, the laser beam will change the angle of deflection, detectable by the photodiode. From the intensity change on the diode segments one can calculate the height difference of the material.

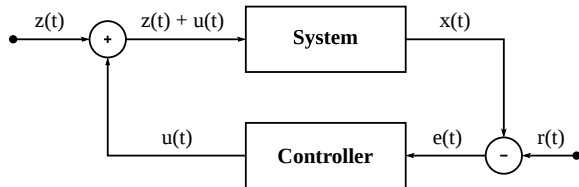


Figure 3: Principle of a feedback loop with disturbance $z(t)$, output $r(t)$, control signal $u(t)$, measured output $x(t)$ and error signal $e(t)$.

2.2 Refinements

Instead of mapping the intensity change on the diode to the force and subsequently to the probe height, often a different approach is used. The tip is set to apply a constant force on the probe. If the force changes, the piezo in z-direction is altered to move the probe to a height where the force is equal to the set point defined before. This has the advantage that the force between tip and probe is approximately constant and therefore it is less likely to damage the sample or the tip. This is ensured via a feedback loop between the the diode and the piezo element as seen in figure 3.

The height information acquired via this method is more strongly connected to the actual sample surface. The mapping is done between the piezo voltage as opposed to the strength of the diode, the angle of the lase beam and the distances between those elements. Therefore introducing less error sources and leading to a more accurate signal.

Relevanz für die Aufnahme der GaSb Proben

3 Data Aquisition

In order to acquire the topology of the samples surface the computer records the z-position in combination with the x- and y-position of the Auswirkungen der PI-Parameter

To optimize the quality of the pictures we can adjust the P- and I-values. These values refer to the proportional and integral-gain of the z-controllers feedback loop respectively. To

For a topographical imaging mode, a feedback loop (Fig. 4.3) is implemented to keep the cantilever deflection constant by changing the tip height (z) while scanning in x and y . In this way, a nearly constant force

is maintained between the tip and sample, and the topographical image is created by recording the voltage applied to the z-piezo as a function of the x and y position. As the tip is scanned, lateral force are

Format

Rauschen- SNR Arten

Methoden und Probleme der Bildbearbeitung

4 Sample Analysis

Features

Rauigkeit der Oberfläche

Aussagen über Wachstumsmethoden

References

- [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): In-situ 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.