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Study of Nanoidentation and Tip Geometry in GAAS (100) at Ultra-Low-Loads for the Patterning of Quantum Dots

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Abstract:

In this study, nanoindentations were produced and characterized for the future patterning of quantum dots. Nanoindentation was performed on a Si-doped (n-type) Vertical Gradient Freeze (VGF) GaAs (100) wafer with a 700 nm GaAs (100) layer grown by molecular beam epitaxy (MBE). Nanoindentation was performed with a Berkovich diamond tip, a cube corner diamond tip, and a 60° conical diamond tip. Nanoindentation of GaAs has been studied in the past, but not at extremely low loads. Previous research has been done on high load (50-200 mN) and low load (200-8000 mN) nanoindentation. The applied load in this study ranges from 400 mN all the way down to 5 mN. The motivation for achieving such low loads is to produce nanoindentations on the same size range as quantum dots (~10-100 nm in width). The smallest indentations achieved were less than 60 nm in width and less than 2 nm deep with the cube corner indenter. The geometry of the indents is characterized using atomic force microscopy (AFM).

Introduction:

The growth of particular combinations of crystalline solids, one atomic layer at a time (epitaxially), can result in the unique ability to form quantum dots. A quantum dot is an extremely small structure (~10-100 nm) that confines electrons in three dimensions, which changes the electronic and optical properties of the material. It is envisioned that these new properties will revolutionize the microelectronics and communications industries, resulting in faster, more powerful, and efficient devices. The formation of quantum dots occurs when thin semiconductor crystalline films buckle due to the stress of having lattice spacing slightly different in size from those of the substrate material. Natural growth of quantum dots results in dots of random size and location on the material substrate. Future devices will require dots with close to identical electronic and optical properties, which are dependent upon the dots possessing equivalent morphology and size, and precise spatial ordering. Thus, it is imperative to learn how to control the positioning and size of quantum dots.

Crude patterning of Indium Arsenide (InAs) quantum dots by scratching and indentation on a Gallium Arsenide (GaAs) substrate has been performed with the tip of an atomic force microscope (AFM). The indentation serves as a nucleation site for the quantum dot to grow. However, the AFM is an imaging tool, which does not allow precision control of (1) the load placed on the surface, (2) tip displacement into the surface, or (3) the loading and unloading rate on the tip. A nanoindenter is an instrument designed to measure material properties on the nanoscale, such as hardness, Young’s modulus, and plastic deformation, and is thus a more promising technique for quantum dot patterning than the AFM.

Before the quantum dot properties can be controlled with this technique, nanoindentation properties at the quantum dot scale (10-100 nm) must be better understood. Nanoindentation of GaAs has been studied in the past, but not at extremely low loads. Previous research has been done on high load (50-200 mN) and low load (200-8000 mN) nanoindentation. However, the loads required to produce sub-100 nm width indentations in GaAs are in a lower regime of <200 mN, which to the authors’ knowledge, is an area that has been studied very little and has not been previously studied using a cube corner or conical indenter. The purpose of this project is to map out the nanoindentation characteristics of a Berkovich, cube corner, and conical diamond nanoindenter tip geometries at ultra-low-loads in GaAs (100).

Experimental:

The TriboIndenter® (Hysitron, Inc.) was utilized for all nanoindentation tests. The sample consists of a VGF epi-ready Si-doped GaAs (100) (supplied by American Xtal Technology (AXT Inc.)) substrate wafer with a 700 nm layer of GaAs (100) grown via molecular beam epitaxy (MBE). MBE growth occurred at 580 °C with a growth rate of 0.69 monolayers per second (ML/s).

Nanoindentations were produced on the sample at room temperature with the following tips: Berkovich diamond (tip radius ~150-200 nm), 90° NorthStar cube corner diamond (tip radius ~53 nm), 60° conical diamond (tip radius <1μm). The following set of indentations were produced on the GaAs (100)
plane at each load with each tip: 5μN, 10μN, 15μN, 25μN, 50μN, 75μN, 100μN, 125μN, 150μN, 175μN, 200μN, 250μN, 300μN, 350μN, 400μN. Load, displacement, and time data was collected for each indentation. Additionally, large marker indentations were produced on the sample at 9000μN to aid in locating the smaller indents. Indentations were placed at a distance of at least ten times the indentation width apart so that strain from adjacent indentations did not affect others. All indentations were performed with a 5 second loading period, followed by a 2 second hold at the peak load and a 5 second unloading period.

Indentations were imaged with a Digital Instruments Nanoscope III atomic force microscope (AFM) at room temperature to analyze the structure of the resulting indentation, and to measure indentation width and depth.

Results and Discussion:

The load versus displacement curve for each indenter tip at a 400 mN peak load is shown in figure 1. It is evident from the three curves that the cube corner indenter places the highest amount of stress on the material, followed by the Berkovich then conical. This is indicated by the slope associated with the loading segment of the curve, in which a higher slope is associated with less stress. This is expected since the cube corner indenter is the sharpest of the tips, therefore supplying less area per amount of force applied. The cube corner and Berkovich tips are both 3-faceted, although the total angle of the cube corner is 90° and the Berkovich tip is 142.3°. The conical tip is in the shape of a cone with a total tip angle of 60°. However, the radius of the conical tip is large (< 1μm) compared to the Berkovich (150-200nm) and cube corner (53nm) tips. Scanning electron microscope images (SEM) of the different tip geometries are shown in figure 2.

Applied stress is an important consideration in the nanoindentation process, because the applied stress must be greater than the critical stress for plastic deformation to be produced. Leipner calculates that the critical stress for dislocation nucleation is ~ 6 GPa6,7. Thus if the tip produces a stress higher than this, plastic deformation and defect nucleation is occurring. Otherwise, the tip will not make a mark in the surface at all. In order to calculate the minimum load required to produce plastic deformation, Hertzian contact theory is utilized, where load as a function of depth varies according to:

\[
\text{Load} = \frac{4}{3} \frac{E^*}{r} \sqrt{rd^3}
\]

Where \( r \) is the tip radius, \( d \) is the depth, and \( E^* \) is the combined tip-substrate modulus. Since the elastic modulus of diamond is much greater than that of GaAs, \( E^* \) is taken as the modulus of GaAs (~ 99.5 GPa)8. Due to the radius on each tip, there exists a load at which the indentation produced will transition from spherical in shape to the geometry of the indenter. It is estimated that the spherical tip radius is all the material experiences until the tip reaches one third of the tip radius of depth into the material during indentation, therefore the width of the indentation and projected contact area can be approximated as:
**Mechanical Engineering:** Robin Prince. Nanoindentation and Tip Geometry

\[
\begin{align*}
W_{\text{Berkovich}}(d) &= W_{\text{Cubic}}(d) = W_{\text{Conical}}(d) = 2\sqrt{(d^2 - 2rd)} \\
\text{for } d < r/3 \\
\text{Area} &= \pi d(2r - d) \quad \text{for } d < r/3
\end{align*}
\]

Utilizing these equations, the lowest load required to produce plastic deformation, the transition load of the indent geometry from spherical to the tip shape, and the smallest indent width and depth can be calculated.

<table>
<thead>
<tr>
<th></th>
<th>Berkovich</th>
<th>Cube Corner</th>
<th>Conical</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lowest Load Required</td>
<td>83.5</td>
<td>8</td>
<td>2720</td>
</tr>
<tr>
<td>(mN)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Transition Load</td>
<td>781.5</td>
<td>226</td>
<td>25500</td>
</tr>
<tr>
<td>(mN)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Minimum Width</td>
<td>133</td>
<td>40</td>
<td>75</td>
</tr>
<tr>
<td>(nm)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Minimum Depth</td>
<td>13.1</td>
<td>4</td>
<td>759.5</td>
</tr>
<tr>
<td>(nm)</td>
<td></td>
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*Table 1 – Calculations of indentation parameters for each tip geometry

However, it was found that indentations (plastic deformation) could be produced at smaller loads than calculated above. The smallest load studied (5 μN) produced a distinct indentation with the cube corner tip, although a rough damaged area was present with the Berkovich tip at 5 μN. Additionally, the indentation shape is clearly in the shape of a triangle at 25 μN load with the cube corner tip and 75 μN load with the Berkovich tip, while at lower loads the shape of the indentation is not well defined.

Pile-up refers to the material that is pushed up above the flat plane of the substrate. It was observed that the cube corner indenter produced a significant amount of pileup at loads ranging from 400 – 25 μN, although the Berkovich tip had an insignificant amount of pile up on all indentations. Figure 4 is an example of the pile-up observed with the cube corner tip and the lack of pile-up observed with the Berkovich tip at a load of 200 μN.

![AFM images of (a) Berkovich nanoindentation at 200 mN with no pile-up and (b) cube corner nanoindentation at 200 mN with pile-up.](image_url)
A transition point from an indentation with significant pile-up to one that sinks into the plane was observed with the cube corner indenter between 25 μN and 15 μN. This is shown in Figure 5 by the AFM cross-section view of the 25 μN indentation and 15 μN indentation. The 25 μN indentation has significant volume of material raised above the plane, while the 15 μN exhibits a sinking-in effect. The varying pile-up as a function of load and tip geometry is an interesting observation because previous literature generally associates pile-up and sinking-in with the ratio of the elastic modulus to the yield stress (E/Y) and the strain-hardening properties of a material. If this were the case, one would expect to see uniformity in piling-up or sinking in for all indenter geometries at all loads, although this is not the what was observed. Further study of this effect must be conducted to determine the cause of variance associated with load and tip geometry.

At loads less than 50 μN the Berkovich ceased to make clear indentations and rather a raised area of material is observed at the indentation site. No indentations were expected to be visible with the conical indenter tip because the load vs. displacement curve generated during loading illustrates that the GaAs recovered completely, as indicated by the loading and unloading imposed on top of each other. No plastic deformation was observed with any of the conical indentations; however, there exists a visible area where the tip made contact with the surface. These areas are represented with a raised area of material a few nanometers in height, similar to the Berkovich indentations at loads less than 50 μN. It is speculated that this type of damage to the surface with no significant indentation produced could be due to the affinity between the diamond tip and the GaAs oxide layer. The stress applied at these points is not enough to produce plastic deformation, although it disrupts the oxide layer and draws the oxide up with the tip as it leaves the surface.

Conclusion:

The geometry of the nanoindenter tip has a significant effect on the type of indentation produced in GaAs (100). The conical indentation does not produce indents at all, but rather a damaged area in the surface at loads less than 400 mN. The Berkovich and cube corner indenters make 3-faceted indentations up to 50 mN and 25 mN loads respectively. Although at loads less than 50 mN with the Berkovich tip there is a raised area of damage at the indentation site similar to what is observed in the conical indentations. The cube corner continues to induce...
plastic deformation and leave indentations at 15 mN, 10 mN, and 5 mN, although the indents are not as reflective of the tip geometry and no material pile-up is observed around the indentations, while significant pile-up is observed in cube corner indentations at loads higher than 25 mN. At high loads with the Berkovich tip, no significant amount of material pile-up is observed around the indentation. Quantum dots are on the order of ~10-100nm in width. Therefore, it is hypothesized that the cube corner indentations at 15 mN and less will be the best size and shape for quantum dot growth, because there is very little pileup and no sharp edges. These indents produced at <15 mN were less than 58 nm in width and 2.5 nm in depth. In order to grow quantum dots at the nanoindentation sites, the indented GaAs wafer would be placed into the MBE chamber and brought up to a temperature around 600°C before growth of the InAs layer. It is hypothesized that the shape of the indentations will change as an effect of annealing at the MBE growth conditions, therefore future work will include annealing of the samples and re-evaluating the nanoindentation properties.

Acknowledgements:

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References:


Faculty comment:

Professor Ajay Malshe, Ms. Prince’s mentor, feels that her research has important implications for further scientific study. In his letter of nomination to the publication board, he wrote:

Ms. Robin Prince is the brightest undergraduate student that I have guided in the last seven years of my faculty career. Robin made her excellent academic and dynamic professional impression because of her passion for scientific curiosity, cutting edge technology and ability to function in a team while maintaining her identity. She has been working on “studying nanomechanics using nanoindentation at tip-GaAs interface” in my research group for the past two years. Her participation is funded through an NSF Research Experience for Undergraduates (REU) program. The project is an interdisciplinary effort among Physics and Mechanical Engineering departments in the field of Nano Integrated Micro Systems.

The investigation on characterizing and controlling damage (i.e. stress, strain, lattice defects) in gallium-arsenide (GaAs) caused by nano-scale mechanical perturbation for the realization of patterned quantum dot arrays is the area of Ms. Robin Prince’s broader scientific interest. Specially, the aim of Ms. Prince’s project and manuscript was to investigate the role of nano indentation tool tip geometry on strain and morphology of the nano imprint patterning of quantum structures. Precision spatial ordering or ‘printing’ of quantum dots by the injection of highly-localized damage sites in 111-V semiconductor surfaces is an area of immense scientific, engineering, and commercial applications interest.

Her approach is to study pattern perturbations in GaAs caused by nanoindentation. The study of nanoscale perturbations in GaAs will give data and clues to the growth mechanisms of quantum dots as a function of the perturbation site’s nanomechanical properties. If this research is successful, it would establish processes for the selective growth of quantum structures and would lead to viable and inexpensive nanomanufacturing processes for fabricating arrays of identical quantum dots. Ms. Prince’s research addresses the following research and education themes: (1) Manufacturing Processes at the Nanoscale, (2) Nanoscale Structures, Novel Phenomena, and Quantum Control, and (3) Nanoscale Devices and System Architecture.