Tunneling Electron Microscopy Investigation Strategy for InAs Nanowires

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Abstract:

We report on methods used for characterization of crystal structure in indium arsenide (InAs) nanowires grown by selective area metal organic vapor phase epitaxy (SA-MOVPE) on gallium arsenide (GaAs) substrates. Using selected area electron diffraction (SAED) and fast Fourier transforms (FFTs) of high-resolution transmission electron microscopy (HRTEM) images, we established appropriate means for nanowire (NW) characterization. We applied these new methods to a series of increasingly silicon-doped InAs wires in order to observe the effects of doping on structure. In addition, we discuss a novel method for HRTEM sample preparation.

	Factor 50	Factor 100	Factor 250	Factor 500
2 port ρ (10^{-5} Ωm)	35	16	7.6	1.8
4 port ρ (10^{-5} Ωm)	16	15	4.4	Not Taken

Table 1: Doping effects on InAs NW resistivity with increasing doping factors.

Introduction:

SA-MOVPE of InAs wires on GaAs substrates yields highly uniform, conductive, and appropriately proportioned NWs for future nanoscale device applications [1]. Our work involved establishing methods for characterizing the crystal structure of our NWs via HRTEM and SAED. In addition to the physical and electrical properties, InAs NWs are receptive to Si doping during growth, and we have observed that increased doping tends to decrease resistivity (Table 1).

Our work continued by investigating the effect doping had on crystal structure in order to potentially establish a correlation between structure on a nanoscale and electrical properties of nanowires.

Growth:

We spun and baked a layer of HSQ onto a GaAs wafer. Next, we spun on an electron beam resist and patterned our wafer with arrays of 50 nm diameter holes at various pitches with electron-beam lithography. After developing the resist, we etched the patterned holes through the HSQ layer, re-exposing the GaAs substrate. Finally, we cleaned the HSQ surface and grew the array of InAs wires by MOVPE.

Nanocrystal Structure:

In III-V nanowires, we see an alternation between wurtzite and zinc-blende (ZB) stacking patterns, whereas bulk growth of III-V materials is typically ZB [2]. The understanding and correlation between crystal structure and electrical transport properties is weak, and to our knowledge, this work is the first attempt to establish a relationship between nanowire doping and crystal structure.

Wurtzite structures stack in a hexagonal close packing pattern, while ZnS crystals follow cubic close packing. The presence of both structures in the wires leads to stacking faults, visible as streaks in HRTEM and SAED. Each structure produces a unique diffraction pattern, and it is critical to understand that the orientation angle of the sample in the tunneling electron microscope (TEM) determines the zone axis, and therefore, the structure we see.

As the angle changes, the zone axis and corresponding diffraction pattern also shift. Convenient relationships exist between low-indexed zone axes of wurtzite and ZnS structure (Table 2) and these relationships are useful for identifying and anticipating crystal structure.

HRTEM Sample Preparation:

Previously, we removed our wires from the substrate via

	0 °	20 °	30 °	40 °	60 °
Wurtzite	[1 0 0]	[3 1 0]	[2 1 0]	[3 2 0]	[1 1 0]
Zinc-blende	[0 1 1]	[-1 3 2]	[-1 2 1]	[-2 3 1]	[-1 1 0]

Table 2: Wurtzite and zinc-blende zone axis relationships.

mechanical force, and placed them onto TEM grids. The result was often large mounds of wires and no selectivity. We began investigating focused ion beam (FIB) methods to create ultra-thin slices of wires. We coated our nanowire arrays with carbon and tungsten. These two materials are highly conductive, but have much different atomic weights, resulting in high contrast in TEM images. After coating, we sliced the samples with FIB in order to isolate and test a small strip of nanowires. This process included the substrate, which was beneficial for confirming the zone axis and orientation angle.

HRTEM and SAED Results:

FFTs of HRTEM images produce reciprocal space images that aid in identification of crystal structure when compared to diffraction pattern simulations for perfect crystal structures, while SAED images are direct diffraction patterns of the sample. FFT and SAED images focus on much different areas, approximately 0.001 μm^2 and 0.031 μm^2, respectively, in our samples. Therefore, FFT is useful for the analysis of structural transitions while SAED is useful for predominant structural characterization. Stacking faults become evident via streaking between FFT or SAED spots.

Our first investigation involved confirming the rotational effects on the diffraction patterns in our wires. We selected a wire with unknown crystal structure (stacking faults confirmed with HRTEM), rotated it 0°, 20°, 30°, and 40° from a reference angle, and we obtained an SAED image at each rotation. When comparing diffraction pattern simulations between wurtzite and ZnS structures, we expected that the 30° rotation of a sample with stacking faults would yield a perfect (no streaks) structure in SAED, and we confirmed this, as seen in Figure 1.

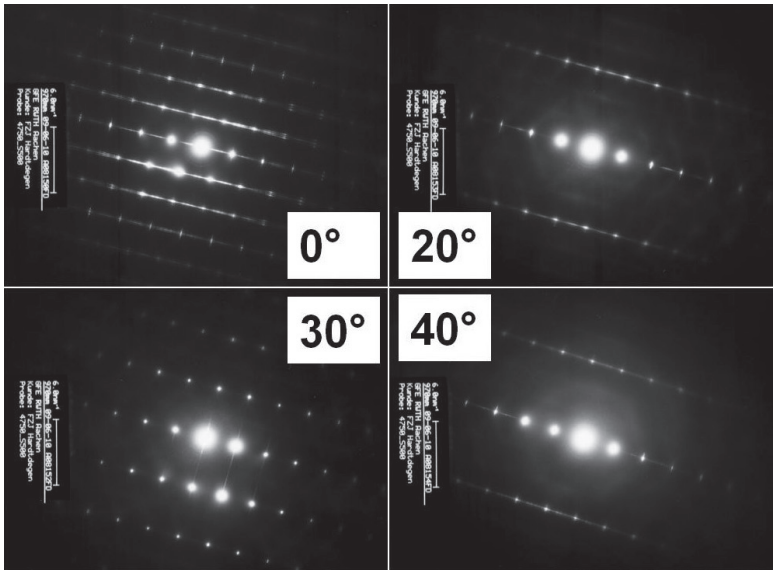

Figure 1: SAED images of rotated sample. Stacking faults (streaks) not visible in 30° rotation.

We then performed a detailed analysis of the diffraction patterns in comparison to wurtzite and ZB simulated patterns. Using ratios representing the distances and angles between particular diffraction spots in the SAED images, we compared these to the ratios seen in the corresponding spots for both wurtzite and ZB simulations. This method allowed us to confidently characterize the predominant crystal structure for this sample was wurtzite.

We next began a new investigation into the effect of doping on crystal structure. Taking four samples of InAs nanowires grown on GaAs (111)B substrates doped at increasing factors, we obtained HRTEM and SAED images of each of the wires. Our first goal was to confirm the zone axes, and therefore the crystal diffraction patterns, we expected to see in the FFTs and SAED. Then, we compared the structures of the wires as doping increased. Our undoped wire showed predominant wurtzite growth, but as the doping factor increased, we began observing more ZB structure in the FFT images. Therefore, we observed doping affecting both electrical properties and structure.

Conclusions:

Our work confirmed the simulated diffraction patterns of both wurtzite and zinc-blende crystal structures via SAED and FFTs of HRTEM images. We established the importance of angle and zone axis when analyzing the samples in the TEM. Finally, we were able to use our characterization method to confidently identify a trend in structural variation due to doping. A continuation of this work may lead to important conclusions regarding doping, structure, and electrical properties of nanowires.

Acknowledgements:

I acknowledge Kamil Sladek, Hilde Hardtdegen, and Martina von der Ahe of Forschungszentrum Jülich, and Thomas Weirich from Aachen University for their many helpful discussions. Thanks to the National Science Foundation and the National Nanotechnology Infrastructure Network International Research Experience for Undergraduates (NNIN iREU) Program for funding.

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