Block course "Templated Indium Arsenide Nanowires"

updated 20010, TC, KC

Our group is pleased to introduce you to the world of Indium Arsenide (InAs) nanowires. InAs is a III-V semiconductor material that is actively researched by many groups. It has interesting electronic properties, including a small band gap and strong spin-orbit interaction (SOI), which makes it a potential candidate material for exotic states of matter. Interestingly, it has been predicted that InAs and other strong SOI materials could be used as building blocks for topological qubits, which has resulted in hundreds of publications in the past couple of decades investigating the properties of various InAs systems. Of particular interest to our group is the characterization of electronic transport parameters in these templated nanowires, such as the mean free path, l_e , and mobility μ .

In this block course we will fabricate a gated InAs nanostructure and perform electrical tests, both at room temperature and at liquid Helium temperature, 4 Kelvin. The main part is the fabrication of the sample, consisting of contacting the templated InAs nanowires (ohmics), creating an isolating layer (ALD) and defining a top gate. The required fabrication procedures are divided into several tasks, which are described in detail in the following pages. You will perform these tasks with the guidance and assistance of the teaching assistant of this course.

If you have any questions at any time (including before or after the block course), please do not hesitate to ask the assistants.

The course is organized with a daily schedule as follows

- 1. Introduction, preparation: eventually start reading
- 2. Clean wafer
- 3. Electron beam lithography: Ohmic contacts
- 4. Native oxide etching
- 5. Deposit ohmics (Ti/Au) and lift off
- 6. Electron beam lithography: ALD window
- 7. Atomic layer deposition and lift off
- 8. Electron beam lithography: Top gate
- 9. Deposit top gate (Ti/Au) and lift off
- 10. Au wire bond
- 11. Test at room temperature
- 12. Test at 4 Kelvin

| | Mon | Tue | Wed | Thu | Fri |
|--------|---------------|----------------|-------------------|----------------|---------|
| Week 1 | # 1 | reading | #2 & #3 (group 1) | #3 (group 2) | #4 & #5 |
| Week 2 | #6 | #7 | #8 (group 1) | $\#8 \pmod{2}$ | #9 |
| Week 3 | #10 (group 1) | #10 (group 2) | #11 | # 12 | report |

1 Intro and preparatory reading

- Introductory discussion

Reading:

Kris Cerveny, "Template-Grown Nanowires for Quantum Transport", Chapter 1

2 Clean wafer

- Clean wafer in acetone and IPA (5 min each)
- Blow dry
- pre-bake on hot plate for 2 min at 120°C
- inspect with optical microscope (take picture)

Reading:

Ralph Williams, "Modern GaAs Processing Methods" chapter 4: Cleaning and Cleanliness, pp. 81-94

3 Electron beam lithography: Ohmic contacts

- Spin electron beam resist (4.5% PMMA), 4000 rpm, ramp speed 4, 40 sec
- Bake on hotplate for 7 min at 180° C
- Inspect with optical microscope
- Add 100 nm Au particles with a toothpick in each corner of the sample
- Inspect with optical microscope:
 - 1. Even coating of resist
 - 2. Measure and note distance from edges to Au particles and markers (take photos)
- Load sample into electron beam machine (e-beam)
- Prepare GDS file (BC_InAs_ohmics.gds)
- Alignment procedure: set focus and angle correction (3-point alignment)
- Write structures: EHT: 30 kV, area dose: 200 μ C/cm², aperture 120 μ m (2 mm field) and 10 μ m (400 μ m field)
- Development in cold developer ((MIBK + IPA):MEK, ratio 100:1:3) at 5°C for 90 sec, rinse with IPA for 30 sec, blow dry
- Inspect under optical microscope (take photos)
- Reactive ion etching (RIE) (gas: 16% O₂, base pressure: 5e-5 mbar, RF power: 30 W, time: 1 min, operating pressure: 250 mTorr

Reading:

Yifang Chen, "Nanofabrication by electron beam lithography and its applications: A review" Microelectronic engineering 135 (2015)

4 Native oxide etch

Attention: NH_4S_x is an acid. Wear appropriate safety gear (labcoat, closed shoes, long trousers, gloves and safety goggles)

This step is time sensitive. Read the instructions carefully and prepare everything before starting the process.

- Prepare two beakers (250 ml) with deionized (DI) water
- Stir NH_4S_x solution for at least 20 min using a magnetic stirrer
- Prepare beaker (20 ml) with 10 ml DI water, put it on hot plate at 40°C, let it equilibrate (min 20 min)
- Prepare two needles, one syringe and one filter for the syringe
- Prepare Sharon (evaporation machine): Check Ti and Au pockets, weigh and record the mass of the Au, take plate to NH_4S_x setup with sticky tape preplaced, leave Sharon door slightly open
- Fill a syringe with 1 ml of NH_4S_x . Turn the syringe upside down and add some air. Remove the needle and attach a new needle with a filter. Remove air from syringe, turn it upside down and push the NH_4S_x through the filter into the beaker with the heated DI water (needs some pressure). Be careful not to stab yourself during the process and hold the needle by the plastic piece in order to prevent it from falling off.
- Immerse the sample in the NH_4S_x solution for 150 sec
- Stop the reaction by transferring the sample into the first 250 ml beaker and softly swirl it around in the water. Do this again in the second beaker. Take the sample out and blow it dry.
- Place the sample on the sample stage plate (on the sticky tape), walk it out of the new clean room and down the hall into the old clean room, then mount it as quickly as possible in the evaporation chamber and start pumping.
- Clean workspace including proper disposal of syringes/needles

Reading:

D. Y. Petrovykh, M. J. Yang and L. J. Whitman, "Chemical' and electronic properties of sulfur-passivated InAs surfaces", Surface Science 523, 3 (2003)
D. B. Suyatin, C. Thelander, M. T. Bjrk, I. Maximov, and L. Samuelson, "Sulfur passivation for ohmic contact formation to InAs nanowires", Nanotechnology 18, 105307 (2007)

5 Deposit ohmics and lift off

- Evaporate Ti (5 nm) and Au (55 nm)
- Let the target cool down, vent the chamber and remove sample from evaporator
- Put the sample into warm acetone $(50^{\circ}C)$ for at least two hours
- Use a plastic pipette to create turbulence until all resist is removed
- If needed, sonicate the sample at 20% for 30 sec. Check for residues and repeat if necessary.
- Rinse with IPA for 1 min, blow dry

- Inspect under optical microscope (take pictures)
- Cleave sample into four individual wafers in a hood with goggles (GaAs microparticles)

Reading:

Rainer Waser, "Nanoelectronics and Information Technology" chapter 8: Film Deposition Methods, pp. 199 - 204 Thomas Heinzel, "Mesoscopic Electronics in Solid State Nanostructures" chapter 4: Experimental Techniques, pp. 101 - 110 (optional)

6 Electron beam lithography: ALD window

- Clean wafer in acetone and IPA (5 min each)
- Blow dry
- pre-bake on hot plate for 2 min at 120°C
- inspect with optical microscope (shoot picture)
- Spin first layer of bilayer electron beam resist (PMMA/MMA 33% 617.08), 6000 rpm, ramp speed 4, 40 sec
- Bake for 5 min at 180° C
- Spin second layer of bilayer electron beam resist (PMMA 679.03), 3500 rpm, ramp speed 4, 40 sec
- Bake for 5 min at 180° C
- Inspect with optical microscope
- Add 100 nm Au particles with a toothpick in each corner of the sample
- Inspect with optical microscope:
 - 1. Even coating of resist
 - 2. Measure and note distance from edges to Au particles and markers (take photos)
- Load sample into electron beam machine (e-beam)
- Prepare GDS file (BC_InAs_ALD.gds)
- Alignment procedure: set focus and angle correction (3-point alignment)
- Write structures: EHT: 10 kV, area dose: 140 μ C/cm², aperture 120 μ m (2 mm field)
- Development in cold developer ((MIBK + IPA):MEK, ratio 100:1:3) at 5°C for 90 sec, rinse with IPA for 30 sec, blow dry
- Inspect under optical microscope
- Reactive ion etching (RIE) (gas: 16% O₂, base pressure: 5e-5 mbar, RF power: 30 W, time: 1 min, operating pressure: 250 mTorr

7 Atomic layer deposition and lift off

- Start ALD machine and preheat the sample stage to 80°C (usually is already done)
- Insert sample and select HfO₂ recipe
- Set heater temperature of sample holder to 200°C(should already be done), ensure the correct amount of layers is set (250).
- Start the process
- Run the purge for HfO₂
- Remove sample when temperature is **below** 90 °C
- Put the sample into warm acetone $(60^{\circ}C)$ for at least two hours
- Use a plastic pipette to create turbulence until all resist is removed
- Sonicate the sample at 20% for 90 sec. Check for residues and repeat if necessary.
- Rinse with IPA for 1 min, blow dry
- Inspect under optical microscope (shoot pictures)

Reading:

S. M. George, "Atomic Layer Deposition: An Overview", Chem. Rev. 110 (2010)

8 Electron beam lithography: Top gate

- Spin electron beam resist (4.5% PMMA), 4000 rpm, ramp speed 4, 40 sec
- Bake on hotplate for 7 min at 180° C
- Inspect with optical microscope
- Add 100 nm Au particles with a toothpick in each corner of the sample
- Inspect with optical microscope:
 - 1. Even coating of resist
 - 2. Measure and note distance from edges to Au particles and markers (take photos)
- Load sample into electron beam machine (e-beam)
- Prepare GDS file (BC_InAs_TopGate.gds)
- Alignment procedure: set focus and angle correction (3-point alignment)
- Write structures: EHT: 30 kV, area dose: 200 $\mu \rm C/cm^2,$ aperture 120 $\mu \rm m$ (2 mm field) and 10 $\mu \rm m$ (400 $\mu \rm m$ field)
- Development in cold developer ((MIBK + IPA):MEK, ratio 100:1:3) at 5°C for 90 sec, rinse with IPA for 30 sec, blow dry
- Inspect under optical microscope (take photos)
- Reactive ion etching (RIE) (gas: 16% O₂, base pressure: 5e-5 mbar, RF power: 30 W, time: 1 min, operating pressure: 250 mTorr

9 Deposit top gate and lift off

- Evaporate Ti (5 nm) and Au (55 nm)
- Let the target cool down, vent the chamber and remove sample from evaporator
- Put the sample into warm acetone $(50^{\circ}C)$ for at least two hours
- Use a plastic pipette to create turbulence until all resist is removed
- If needed, sonicate the sample at 20% for 30 sec. Check for residues and repeat if necessary.
- Rinse with IPA for 1 min, blow dry
- Inspect under optical microscope (take pictures)
- Cleave sample into four individual wafers (one for each participant)

10 Au wire bond

- Cleave sample that it fits into chip carrier and clean it
- Clean and sonicate chip carrier (both in acetone for 5 min)
- Rinse with IPA, blow dry
- Glue sample into chip carrier with a droplet of silver paint (Leitsilber), wait for it to dry
- Set the bonding parameters and bond the sample with Au wire
- Inspect the sample under microscope (take pictures)
- Draw a diagram of the bonding scheme, carefully numbering the wires

Reading:

Thomas Heinzel, "Mesoscopic Electronics in Solid State Nanostructures" chapter 4.1.5: Bonding, pp. 110-111

11 Test at room temperature

- Check electrical wiring of dip stick with cross-linked carrier (1-2, 3-4, etc.)
- Connect everything and ground all wires at the break-out-box
- Place the sample in the sample holder, carefully checking orientation and close the can
- Do two- and four-point I/V measurements

Reading:

Thomas Heinzel, "Mesoscopic Electronics in Solid State Nanostructures" chapter 4.2: Elements of Cryogenics, pp. 110-120 chapter 4.3: Electronic measurements on nanostructures, pp. 121-123, 127-128 online tutorial: http://www.allaboutcircuits.com/vol_1/chpt_8/9.html

12 Test at 4 Kelvin

- Dip sample into liquid helium transfer dewar
- Repeat two- and four-point measurements

13 Report

A report of 2500 to 5000 (do not exceed 5000) words has to be prepared describing the techniques used, the experiments performed and the measurements made. It would be appropriate to have a few pictures of the processed sample at some of the various processing steps. As the language of science is generally English, we highly recommend that your report is written in English. The preferred scientific text processing system for physicists is LaTeX, and we would like to encourage you to prepare your report using LaTeX. Structure the report well. It should include at least the following sections:

- 1. Introduction and Motivation
- 2. Experiments and Results
- 3. Conclusion and Discussion

The report will be graded using the published block course criteria, also available on our webpage.