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| f2_color_pos_EHSS_CMYK | | | STANDARD OPERATING PROCEDURE | | | | | | | | | |
| **Indium gallium arsenide selective etch rates** | | | | | | | | | |
| College/Dept: | | College of Engineering | | | | | Building/Room: | Ruch 105 | | | | |
| Laboratory Name: | | Collaboratory for Epitaxy of Materials | | | | | Revision: |  | | | | |
| Principal Investigator: | | Paul Simmonds | | | | | Author: | Paige Wilson/James Rushing | | | | |
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| Before the work detailed in this procedure may begin, the intended user must read and understand this document.  This document must be approved by the PI, the college’s safety liaison, and EHSS.  Any changes to this document, however minor, must be submitted for approval by the PI, the college’s safety liaison, and EHSS. | | | | | | | | | | | | |
| **Approval** | | | | | | | | | | | | |
| Intended user: | James Rushing | | | |  |  | | | ­­­­­­­ | 11/07/2022 |  | |
| Name, Title | | | |  | Signature | | |  | Date |  | |
| Reviewed and Approved by: |  | | | |  |  | | |  |  |  | |
| Name, Title | | | |  | Signature | | |  | Date |  | |
| Paul Simmonds, PI | | | |  |  | | | ­­­­­­­ | 11/07/20222 |  | |
| Name, Title | | | |  | Signature | | |  | Date |  | |
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| Name, Title | | | |  | Signature | | |  | Date |  | |

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| **Overview** |
| This SOP describes a process for measuring the selective etch rates of InGaAs from InAlAs that are both lattice matched to InP for different etchant chemistries. We will use this approach to selectively remove the epitaxial InGaAs layer from InAlAs. |
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| **Scope** |
| This SOP covers:   * Covering sample with photoresist * Preparing etching solution and etching * Sample height measurement |

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| **Potential Hazards** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| ☒ | | Chemical | | ☒ | | | | Thermal | | | ☐ | Hydraulic | | | | | | | | ☐ | Electrical | | | ☐ | | | | Slip/Trip | | ☐ | Biological |
| ☐ | | Mechanical | | ☐ | | | | Radiation | | | ☐ | Pneumatic | | | | | | | | ☐ | Fire | | | ☐ | | | | Fall | | ☐ | Other |
| **Hazard Specifics:** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Succinic Acid is corrosive and causes irritation to eyes and skin, respiratory tract, and may cause digestive tract irritation.  Methylsuccinic Acid is corrosive and causes irritation to eyes and skin, respiratory tract, and may cause digestive tract irritation  Dimethylsuccinic Acid is corrosive and causes irritation to eyes and skin, respiratory tract, and may cause digestive tract irritation  Citric Acid may form combustible dust concentrations in air and can cause serious eye irritation  Hydochloric Acid may be corrosive to metals and causes severe skin burns and eye damage  Hydrogen Peroxide may cause fire or explosion; strong oxidizer. Causes severe skin burns and eye damage. Harmful if swallowed or if inhaled.  Ammonium Hydroxide causes severe skin burns and eye damage. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
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| **Engineering Controls (EC)** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| ☒ | | Fume hood | | | | | ☐ | | | Biosafety Cabinet | | | | | | | ☐ | | Other Local Exhaust | | | | | | ☒ | | | Shielding | | ☐ | Other |
| EC Specifics: | | | | | |  | | | | | | | | | | | | | | | | | | | | | | | | | |
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| **Training Requirements** – except for classroom lab safety, must be completed prior to performing the procedure | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| ☒ | | Online Laboratory Safety Training (MEC 104) | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
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| **Personal Protective Equipment (PPE)** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| ☐ | Safety glasses | | | | ☐ | | | | Safety goggles | | | | | ☐ | Face shield & safety glasses | | | | | | | | | | | ☒ | | | Face shield & safety goggles | | |
| ☒ | Lab coat | | | | ☒ | | | | Apron | | | | | ☐ | Tyvek suit | | | | | | | | | | | ☐ | | | Tyvek sleeves | | |
| ☒ | Gloves | | | | ☒ | | | | Leg coverings | | | | | ☐ | Hard hat | | | | | | | | | | | ☐ | | | Hearing protection | | |
| ☐ | Respirator | | | | ☒ | | | | Shoes | | | | ☐ | | Fall protection | | | | | | | | | | | | ☐ | | Other | | |
| PPE Specifics: | | | | | Bunny suit that covers the whole body as well as booties to cover shoes and acid gloves when handling acids. | | | | | | | | | | | | | | | | | | | | | | | | | | |
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| **Equipment, Materials, Supplies, & Facility Requirements** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Glassware, DI water, hydrogen peroxide, ammonium hydroxide, succinic acid, dry nitrogen gas, cleanroom wipes, tweezers. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
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| **Handling, Work Area & Storage Requirements** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Fume hood, storage for acids, disposal for waste acid solutions  In case of leak or spill, contact the Laboratory PI, Peter Miranda, cell phone number is 208-426-5713,  and Lab Manager Travis Gabel, cell phone number is 208-426-5752,  and EHSS (24/7 line 208-863-8024).  Storage: Keep container tightly closed. Keep in a cool, well-ventilated place. Highly toxic or infectious materials should be stored in a separate locked safety storage cabinet or room. It will be stored in Ruch 105 on the shelf for “Moderately Hazardous Chemicals”. Keep container tightly closed.  **General Safety Procedures**   1. All containers should be properly labeled, including chemical name, date, name of owner, and chemical safety label information. 2. After handling chemicals, washing hands is mandatory before eating or leaving the lab area. 3. Copies of the SDS for all chemicals described in this protocol must be kept in Ruch 105 and any other lab used for storage and mixing of these chemicals.   All waste containing, or contaminated with, hazardous chemicals must be stored in closed container in the chemical waste collection area. To have chemical waste picked up, fill out a Waste Pickup Form (UNH-Chem, an online system.) E-mail [christophersiepert@boisestate.edu](mailto:christophersiepert@boisestate.edu) for any questions or concerns.  Solid waste (such as used gloves, pipette tips, and vials, etc.) contaminated with low potential hazard chemicals can be disposed of in the chemical waste bin. When the chemical waste bin is full, the bag can be sealed and put in the normal trash.  **General Handling and Storing for all chemicals.**  *Handling*  Wear personal protective equipment. Ensure adequate ventilation. Use spark-proof tools and explosion-proof equipment. Avoid contact with skin, eyes and clothing. Avoid ingestion and inhalation. Keep away from open flames, hot surfaces and sources of ignition. Take precautionary measures against static discharges.  *Storage*  Keep containers tightly closed in a dry, cool and well-ventilated place. Keep away from heat and sources of ignition. Will be stored in Ruch 105 under the fume hood in the storage area for “Flammable Chemicals”. Keep container tightly closed. Keep away from heat. Keep away from sources of ignition. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| **Emergency Response Equipment & Supplies** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| ☒ | Eyewash | | | | ☐ | | | | Fire extinguisher | | | | | | | ☒ | | First aid kit | | | | ☐ | Calcium gluconate gel (HF use) | | | | | | | | |
| ☒ | Safety shower | | | | ☐ | | | | [Fire blanket](http://www2.boisestate.edu/ehs/EHS%20Policies-Programs/Spill%20Kit%20Supplies%20-%20General%20Laboratory.pdf) | | | | | | | ☒ | | Spill kit | | | | ☐ | Emergency gas shutoffs | | | | | | | | |
| ☐ | Drench hose | | | | ☐ | | | | Other: | | | | | | | | | | | | | | | | | | | | | | |
| Description: | | | Use spill kit in cleanroom as per manufacture instructions. | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
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| **Spill Response** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| *Spill*:  For liquid: Soak up with inert absorbent material and place in container for disposal. Keep in suitable, closed container for disposal in the chemical waste collection area. Do not let product enter drains.  For powders: Sweep up and place in container for disposal. Avoid raising dust. Ventilate area and wash spill site after material pick up is complete. DO NOT empty into drains. DO NOT touch spilled material.  Ensure to contact the Laboratory PI, Peter Miranda, cell phone number is 208-426-5713, and Lab Manager Travis Gabel, cell phone number is 208-426-5752, and EHSS (24/7 line 208-863-8024). An incident report should be filed according to safety policy. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
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| **Decontamination & Waste Disposal** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Dilute etching solution and store in an approved waste container with a vented lid.  *Waste:* Dispose according to all applicable regulations. May be harmful to aquatic life. Can be dangerous if allowed to enter drinking water intakes. Do not contaminate domestic or irrigation water supplies, lakes, streams, ponds, or rivers.  All waste containing, or contaminated with, hazardous chemicals must be stored in closed container in the chemical waste collection area. To have chemical waste picked up, fill out a Waste Pickup Form (UNH-Chem, an online system.) E-mail [christophersiepert@boisestate.edu](mailto:christophersiepert@boisestate.edu) for any questions or concerns.  Solid waste (such as used gloves, pipette tips, and vials, etc.) contaminated with low potential hazard chemicals can be disposed of in the chemical waste bin. When the chemical waste bin is full, the bag can be sealed and put in the normal trash. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |

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| **Additional Safety Information** | | |
| Review of applicable safety references such as material safety data sheets to ensure appropriate protective measures, spill supplies, and first aid procedures.  First Aid: General for all chemicals  *If inhaled* Allow the victim to rest in a well ventilated area. Seek immediate medical attention.  *In case of skin contact* After contact with skin, wash immediately with plenty of water. Gently and thoroughly wash the contaminated skin with running water and non-abrasive soap. Be particularly careful to clean folds, crevices, creases and groin. Cover the irritated skin with an emollient. If irritation persists, seek medical attention.  *In case of eye contact* Flush eyes with running water for at least 15 minutes, keeping eyelids open. Cold water may be used. Do not use an eye ointment. Seek medical attention  *If swallowed* Do not induce vomiting. Examine the lips and mouth to ascertain whether the tissues are damaged, a possible indication that the toxic material was ingested; the absence of such signs, however, is not conclusive. Loosen tight clothing such as a collar, tie, belt or waistband. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek immediate medical attention.  If first aid or medical attention is needed for someone in the lab, ensure to contact the Laboratory PI, Peter Miranda, cell phone number is 208-426-5713, and Lab Manager Travis Gabel, cell phone number is 208-426-5752, and EHSS (24/7 line 208-863-8024). An incident report should be filed according to safety policy.  Fire Measures  Extinguishing Media: Water fog in flooding quantities. Apply water from as far a distance as possible.  Fire Fighting Methods and Protection: Firefighters should wear full protective equipment and NIOSH approved self-contained breathing apparatus.  Fire and/or Explosion Hazards: Avoid Dusting. May become explosive when dispersed in air.  Water sprayed onto burning material may cause frothing and splattering of burning liquid with possible injury of firefighting personnel. Fire or excessive heat may produce hazardous decomposition products.  If a fire occurs in your lab, ensure to contact the Laboratory PI, Peter Miranda, cell phone number is 208-426-5713, and Lab Manager Travis Gabel, cell phone number is 208-426-5752, and EHSS (24/7 line 208-863-8024). An incident report should be filed according to safety policy. | | |
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| **References** | | |
| A.R. Clawson. Guide to references on III-V semiconductor chemical etching, Department of Electrical and Computer Engineering, University of California at San Diego, 27 August 2000.  Liu, Z., Sun, Y., Machuca, F., Pianetta, P., Spicer, W. E., & Pease, R. F. W. (2003). *Optimization and characterization of III–V surface cleaning. Journal of Vacuum Science & Technology B: Microelectronics and Nanometer Structures, 21(4), 1953.*  Hydrochloric Acid, Fisher Scientific, <https://preview.fishersci.com/store/msds?partNumber=A144S500&productDescription=HYDROCHLORIC+AC+SAFECOTE+500ML&vendorId=VN00033897&countryCode=US&language=en>  Citric Acid, Fisher Scientific, <https://www.fishersci.com/store/msds?partNumber=A104500&productDescription=CITRIC+ACID+GRAN+CERT+ACS+500G&vendorId=VN00033897&countryCode=US&language=en>  Methyl Succinic Acid, ThermoFisher Scientific,<https://assets.thermofisher.com/DirectWebViewer/private/document.aspx?prd=ALFAAH60967~~PDF~~MTR~~CLP1~~EN~~2021-01-13%2001:36:37~~Methylsuccinic%20acid~~>  Dimethyl Succinic Acid, Fisher Scientific, <https://www.fishersci.com/store/msds?partNumber=AAB2439403&productDescription=22-DIMETHLSUCCINIC+ACID+99%25+1G&vendorId=VN00024248&countryCode=US&language=en>  Succinic Acid, Fisher Scientific, <https://fscimage.fishersci.com/msds/95199.htm>.  Ammonium Hydroxide, Fisher Scientific, <https://fscimage.fishersci.com/msds/00211.htm>.  Hydrogen Peroxide, Fisher Scientific, <https://fscimage.fishersci.com/msds/11189.htm>. | | |
| **Procedure** | | |
| **STEPS** | | |
|  | **Potential Hazards** | **EC, Haz. Mitigation Device, PPE** |
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| 1. **Prepare and mix relevant etching solution so that it comes to room temperature before you are ready to use it**    * 1. Create succinic acid etch solution by adding 100 grams dimethyl succinic acid to 1 liter deionized water. Mix a (25:1) dimethyl succinic acid solution to H2O2 in a glass beaker big enough to fit the wafer sample. Use two graduated cylinders, one labeled acid for the succinic and one for the H2O2. First measure and pour the succinic acid into the glass beaker then measure and pour the H2O2 into the succinic acid while avoiding any splashing. Mix carefully. Bring pH to 5.5 [PW1] by slowly adding NH4OH.      2. Prepare a glass beaker with DI water for submerging the samples after each etch. | **Hazards**: Dimethyl Succinic Acid is corrosive and causes irritation to eyes and skin, respiratory tract, and may cause digestive tract irritation.  Hydrogen Peroxide may cause fire or explosion; strong oxidizer Causes severe skin burns and eye damage. Harmful if swallowed or if inhaled.  Ammonium Hydroxide causes severe skin burns and eye damage. | **PPE**: Gloves, Shoes, bunny suit, booties, goggles/glasses, and face shield at all times. |
| 1. **­Coat front of sample (i.e. InGaAs) with photoresist.** 2. Spin the photoresist onto InGaAs surface (negative resist is preferable but positive resist will also work) following manufacturer’s instructions. 3. Follow the JST Solvent Bench to set up the spin coat station of HDMS and photoresist. Write down the recipe for each material 4. Verify the temperature of the hot plate is 115C 5. Set up the recipe for ECE540L-HMDS 6. Dispense 1.5 – 2ml of HMDS per wafer using a clean pipette. Softbake the HDMS on the hot plate for 60 sec. at 115C 7. Set up the spin coat recipe for ECE540L-SPR220-3.0 8. Follow the same process for dispensing the PR onto the wafer. Be careful not to generate any bubbles in the PR film since they will impact the coating quality of the PR. DO NOT USE THE SAME PIPETTE FOR PR. 9. Softbake the PR on the hot plate for 90 sec. at 115C 10. Measure the PR thickness using the ellipsometer 11. Use recipe for ECE540K (THIS MIGHT BE A SILICON RECIPE NEED TO LOOK INTO IT) for a map scan of your spun on PR. You should achieve a thickness of about 1.8 microns. If not, you’ll need to rework the wafer PR again. | **Hazards**: Photoresist is a combustible liquid and vapor and causes irritation to eyes, nose, and respiratory tract. Prolonged, repeated contact, inhalation, ingestion, or absorption through the skin, may cause toxic effects to internal organ systems. | **PPE**: Gloves, Shoes, bunny suit, booties, goggles/glasses, and face shield at all times. |
| 1. **Use straight edge of silicon wafer to cover half of InGaAs surface.**    * 1. Expose to UV light in mask aligner according to manufacturer instructions. | **Hazards**: Photoresist is a combustible liquid and vapor and causes irritation to eyes, nose, and respiratory tract. Prolonged, repeated contact, inhalation, ingestion, or absorption through the skin, may cause toxic effects to internal organ systems. |  |
| 1. **Develop photoresist and cure according to manufacture instructions.** | **Hazards**: Photoresist is a combustible liquid and vapor and causes irritation to eyes, nose, and respiratory tract. Prolonged, repeated contact, inhalation, ingestion, or absorption through the skin, may cause toxic effects to internal organ systems. |  |
| 1. **Measure height of photoresist step on InGaAs surface.**    * 1. Use profilometer to measure resist height 4 times in different locations and take the average.      2. Subtract this initial photoresist height from future measurements to obtain actual etch depth. |  |  |
| 1. **Clean wafer with pre-etch solution.** 2. Etch wafer in HCL: H2O (1:3) solution for 1 min to deoxidize the surface. | **Hazards**: Hydrochloric Acid may be corrosive to metals and can cause severe burns to the skin and eye damage. |  |
| 1. **Etch InGaAs.**    * 1. Choose a small interval of time to begin with (i.e. 5 minutes) Using plastic tweezers completely submerge sample in etching solution.      2. Agitate the sample or use a magnetic stirrer during etching to ensure constant etch rate.      3. After time has elapsed, immediately immerse and swirl sample in DI water for 30 seconds to stop etching. Blow dry with nitrogen gas. | **Hazards**: Dimethyl Succinic Acid is corrosive and causes irritation to eyes and skin, respiratory tract, and may cause digestive tract irritation.  Hydrogen Peroxide may cause fire or explosion; strong oxidizer Causes severe skin burns and eye damage. Harmful if swallowed or if inhaled.  Ammonium Hydroxide causes severe skin burns and eye damage. | Bunny suit that covers the whole body as well as booties to cover shoes and acid gloves when handling acids. |
| 1. **Measure height of photo resist again.** 2. Use profilometer to remeasure resist height 4 times in different locations and take the average. 3. Subtract the initial photoresist height from this new height measurement to obtain actual etch depth. 4. Plot data point on graph of total etch depth vs. total etch time. |  |  |
| 1. **Repeat steps 6 & 7, plotting etch depth vs. etch time data after each iteration.** | **Hazards**: Succinic Acid is corrosive and causes irritation to eyes and skin, respiratory tract, and may cause digestive tract irritation.  Hydrogen Peroxide may cause fire or explosion; strong oxidizer Causes severe skin burns and eye damage. Harmful if swallowed or if inhaled.  Ammonium Hydroxide causes severe skin burns and eye damage. | Bunny suit that covers the whole body as well as booties to cover shoes and acid gloves when handling acids. |
| 1. **Look carefully for a significant change in etch rate**    * 1. We expect the etch rate will reduce significantly once the InGaAs has been completely removed and the InAlAs is exposed.      2. You will see this as an abrupt change in the slope of the etch depth vs. etch time graph.      3. Once this occurs, repeat steps 6 & 7 two more times to measure the etch rate of the InAlAs. | **Hazards**: Succinic Acid is corrosive and causes irritation to eyes and skin, respiratory tract, and may cause digestive tract irritation.  Hydrogen Peroxide may cause fire or explosion; strong oxidizer Causes severe skin burns and eye damage. Harmful if swallowed or if inhaled.  Ammonium Hydroxide causes severe skin burns and eye damage. | Bunny suit that covers the whole body as well as booties to cover shoes and acid gloves when handling acids. |
| 1. **Waste Disposal** 2. Carefully transfer etching solution to a larger beaker and slowly add sodium carbonate until a litmus test shows a pH of 5-7 3. Store neutralized waste into an approved and labeled waste container with a vented lid. 4. Rinse each beaker and cylinder with DI water and make sure all chemicals are in their proper location. | **Hazards**: Succinic Acid is corrosive and causes irritation to eyes and skin, respiratory tract, and may cause digestive tract irritation.  Hydrogen Peroxide may cause fire or explosion; strong oxidizer Causes severe skin burns and eye damage. Harmful if swallowed or if inhaled.  Ammonium Hydroxide causes severe skin burns and eye damage. | Bunny suit that covers the whole body as well as booties to cover shoes and acid gloves when handling acids. |
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