SAFE WORK PROCEDURE:

Metal Organic Chemical Vapor Deposition system-MOCVD (Aixtron)

Doc No. SWP-COE-11
Last Rev/Date 00 30 Mar 2010
Current Rev/Date 00 30 Mar 2010
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1. **Application to:**

   All personnel working on the Aixtron MOCVD 200/4 system 1442 (GaAs and InP based materials growth)

   Reference: Specify list of Literatures/ Standards/ Codes of Practice

   i. MOCVD operation procedure
   ii. MOCVD emergency shutdown procedure
   iii. MOCVD shutdown procedure
   iv. (References to Attachment)

2. **Pre-requisite for Operation of Aixtron MOCVD:**

   2.1. Must have attended the operation and safety training.
   2.2. Must be fully understand the system risk assessment.
   2.3. Must have read and understood the operation procedure
   2.4. Must have apprenticed in using the system together with experience user of the system
   2.5. Must be able to change gas cylinders and gas tanks when they are depleted

**Process Management**

2.1 **Brief Description of the Process/ Operation**

1) MOCVD GaAs/InP based material growth using MO sources and flammable gas such as silane- SiH4 and Hydrogen-H2

Location of Operation/ Process: **COE, Clean room, NUS, E3-03-09**
Lab Name/ Unit Number of Lab

2.2 **Safety Precautions for Normal Course of Work**
*(Specify any critical operating steps/ parameters for safe operation, if any)*

1) Follow the operation manual for MOCVD growth
2) Follow the vendor manual

2.3 **Safety Precautions during an Emergency**
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(Specify any critical operating steps/ parameters for emergency operation/ shut down, if any)

1) Follow the emergency showdown procedure for MOCVD growth
(Refer to flow chart attachment 6.3)

3. Hazards that may be present:

3.1. Handling of hot wafer carrier may burn hand.
3.2. Hot furnace may cause burns.
3.3. Potential hazard of leakage of Metal-organic gases, hydrogen, nitrogen, silane or ammonia gases from system.
3.4. Micro size carbon particles may cause breathing problem when inhaled while opening growth chamber.

Proper Monitoring/ Control

3.1 List of Hazardous Materials(if used/ if any)*
*Attach all relevant MSDSs

<table>
<thead>
<tr>
<th>Name</th>
<th>Hazard Property</th>
<th>TLV/PEL</th>
<th>STEL</th>
<th>Ceiling</th>
<th>IDLH</th>
<th>LD50/LC50 (Acute toxicity)</th>
<th>TWA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tertiarybutylphosphine-TBP (C4H11P)</td>
<td>Highly Flammable</td>
<td></td>
<td>N.E.</td>
<td></td>
<td>N.E. (LD50), (LC50) 510ppm(4h) or 1144ppm(3h) no lethality (&gt;1000ppm)</td>
<td>0.5mg/m3 (8h)</td>
<td></td>
</tr>
<tr>
<td>Tertiarybutylarsine-TBAs (C4H11As)</td>
<td>Highly Flammable / Very Toxic</td>
<td></td>
<td>N.E.</td>
<td></td>
<td>N.E.(LD50), (LC50) 90ppm (4h) (140ppm)</td>
<td>0.5mg/m3 (8h)</td>
<td></td>
</tr>
<tr>
<td>Silane-SiH4 (1% in H2)</td>
<td>Flammable/ Explosive</td>
<td></td>
<td></td>
<td></td>
<td>Simple asphyxiant gas, No occupational exposure limits established.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hydrogen -H2</td>
<td>Flammable gas, may cause flash fire</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**NE = Not Established  ***LC50= Inhalation / LD50=Oral  ****TWA= Time Weighted Exposure Limit
3.2 Waste Discharge/ Waste Disposal (if any)

1) Specify possible wastes/ contaminants discharged by the process (if any)
   - Phosphorus/arsenic was neutralized by H2SO4+ Na2BrO3 (first stage) and NaOH (second stage) solutions
   - III-V compounds by combining Ga, In, Al and P, As, trapped in the particle trap.

2) Specify special disposal method or any prior treatment is required (if any)
   - Qualified vendor collects the used scrubber solutions and carry out the subsequent treatment.

3.3 Labeling/ Warning Sign

The PI/ Supervisor/ Staff-in-charge shall put up the following warning signs at designated/ prominent areas.

1) MOCVD reactor
2) Gas cabinets

4. Personal Protection Required:

Provisions of Personal Protective Equipment (PPE)

List of Recommended PPE to Persons Carrying Out the Work

<table>
<thead>
<tr>
<th>Personal Protective Equipment (PPE)</th>
<th>Type of Activity/ Work</th>
</tr>
</thead>
<tbody>
<tr>
<td>Respirator/ goggles</td>
<td>Reactor maintenance</td>
</tr>
<tr>
<td>Respirator/acid resistant gloves,</td>
<td>Scrubber maintenance</td>
</tr>
<tr>
<td>goggles (when necessary)</td>
<td></td>
</tr>
<tr>
<td>Respirator/ Leak-test equipment/</td>
<td>Change MO sources</td>
</tr>
<tr>
<td>goggles</td>
<td></td>
</tr>
</tbody>
</table>

*Before any prior activity/work begins main system should be shut into N2 line purge*
5. Procedures:

**MOCVD Growth Chamber loading/unload Process Flow Chart**

- **Wafer loading**
  - Refer to appendix 1 on MOCVD Growth Process Flow Chart

1. **Open ante-chamber door**
   - Yes
   - **Load substrate into ante-chamber**
   - No

2. **Close antechamber door and evacuate/refill the antechamber 3 times**
   - Yes
   - **Open reactor gate**
   - **Load substrate into reactor**
   - **Close reactor gate**
   - **Open related MO/gas valves**

3. **Run growth recipes**
   - Yes
   - **Unload the wafer from reactor**
   - **Transfer structures to load lock**
   - **Evacuate load lock and fill nitrogen**
   - **Open load lock**

**Appendix 2**
5.2 Maintenance of the MOCVD growth (Aixtron 200/4 System 1442)

5.2.1 Refer to Aixtron 200/4 system 1442 operation Manual Chapter -7 Maintenance and repair.

5.3 Specific Processes

5.3.1 First Aid and Emergency Procedure

5.3.2 In case of explosion/ fire/ spillage/ gas leakage, the PI/ Supervisor/ Staff-in-charge shall alert all staff & students working in the lab to evacuate to a safe area immediately.

5.3.3 The PI/ Supervisor/ Staff-in-charge shall activate the Incident Response Team (IRT) by calling 6874-4154 (IMRE) from a safe area.

5.3.4 The Incident Response Team (IRT) from NUS shall take necessary control measures to contain the situation and protect the staff/ students involved as well as the environment.

5.3.5 The PI/ Supervisor/ Staff-in-charge shall investigate the incident/ accident and submit the “EHS Incident Report/ IMRE Accident Report” within 24 hours to IMRE and NUS.

6. Communication Frequency:

6.1 The PI/ Supervisor/ Staff-in-charge shall inform/ brief the staff/ students (involved the process) of the risk assessment done and established safe work procedures, including the appropriate measures to reduce or eliminate the hazards to be communicated as & when required and every once a year.

6.2 The PI/ Supervisor/ Staff-in-charge shall train the staff/ students involved in the process on the DOS & DON'TS (see attached 6.1).
6.3 The PI/ Supervisor/ Staff-in-charge shall issue proper PPE to the staff/ students involved in the process.

6.4 List of Other Trainings Required:

1) Training on the usage of gas lines and gas cabinet
2) Training on the operation of MOCVD system

7. Attachment

7.1 MOCVD Do’s & DON’TS
7.2 Specify list of Literatures/ Standards/ Codes of Practice
7.3 MSDS on Hazardous Materials

Prepared by: Mr. Wang Ben Zhong / Mr. Jack Eng

Approved by: Prof Chua Soo Jin

**Attachment 7.1**

**MOCVD Do’s AND DON’TS**

<table>
<thead>
<tr>
<th>S/N</th>
<th>Do’s</th>
<th>DON’TS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ask your supervisors about the safety precaution/ risk involvement/ first aid procedure etc before start work.</td>
<td>Never start MOCVD process when the system status label is not in run state (in service state)</td>
</tr>
<tr>
<td>2</td>
<td>Always follow the warning sign, and put on all the required PPE.</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>Always follow the operation procedure</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Check the conditions of the system from time to time during growth</td>
<td></td>
</tr>
</tbody>
</table>
Attachment 7.2

Reference Specify list of Literatures/ Standards/ Codes of Practice

Aixtron Operation Manual (Aix 200/4 Project1442)

- MOCVD operation procedure – Chapter.1-7
- MOCVD Start-up of the system – Chapter.5 Page.13-19 (flow chart)
- MOCVD System shut off procedure – Chapter.5 Page.20-21 (flow chart)
- MOCVD Emergency shutdown procedure- chapter.6 Page.10-11 (flow chart)

- MDA Scientific CM4 four-point continuous monitor
  - Appendix A Specification document
  - Appendix C- Chemcassette detectable Gases

*MOCVD= Metal Organic chemical vapor Deposition*

Attachment 7.3

Material safety data sheet (MSDS) on Hazardous Materials

- Tertiarybutylphosphine-TBP
- Tertiarybutylarsine-TBAs
- Silane-SiH4 (1% in H2)
- Hydrogen-H2

Remarks:

Metal organic chemical vapor deposition (MOCVD) process is using TMGA and TMIn which is a liquid or solid phase organ-metallic compound, the process also uses Tertiarybutylarsine-TBAs is alternate arsenic
(AsH3) source with its chemical properties of lower vapor pressure characterized thus is safer to handle than arsenic (AsH3).

As Tertiarybutylarsine-TBAs compound is been use in MOCVD growth process within the reactor growth chamber at the rate of 500-610 degrees C temperature it decomposition with arsenic residue which draw into exhaust gas detoxification by wet chemical scrubbing process where arsenic/Phosphorus was neutralized by H2SO4+ Na2BrO3 in first stage following with NaOH second stage solution and the materials of V and its compound of III-V are trapped in the particle trap.

**Safety action:**

On above listed Hazardous Materials only Arsenic and its Compounds (such as TBAs) are listed in the WSH (Medical Examinations) Regulations and WSH (General Provisions) Regulations. Below stated requirement is needed under the WSH Regulations. (http://www.mom.gov.sg/publish/momportal/en/communities/workplace_safety_and_health/maintaining_a_safe_workplace/health_and_environmental.html)

- Conduct Environmental Surveillance (/ Air Monitoring)

- Prescribed Hazards requiring medical examinations under the workplace safety and Health (medical examinations) regulation by MOM

Conduct Environmental Surveillance (/ Air Monitoring) completed by Setsco services Pte Ltd on 22/09/2008(base-line test) and 11/11/2008(final test) attachment with test report document En17667/LKM and EN18818/LKM. Other additional material Tributylphospate, arsenic, aluminium, zinc oxide, indium and carbon black etc was included in this Air Monitoring test.

**Addition information of MSDS on Arsine (AsH3) and Phosphine (PH3) for comparison differences on Tertiarybutylarsine (TBAs) and Tertiarybutylphosphine (TBP)**