Hazard Controls: HPM Chemicals

Presented by:
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Steve Trammell, EORM
Matt Wyman, KFPI
Overview

- What are HPM Liquids and Solids?
  - Bulk Chemical
    - Any process chemical needed in quantities over multiple gallons/pound/kilograms
  - What forms do they come in?
    - Liquids
    - Slurries
    - Powders
    - Particulate
Overview

- What is Bulk Chemical Delivery?
  - Bulk Chemical Delivery System Elements
    - Enclosure
    - Bulk Container
    - Deliver Control System
    - Piping and Valves
    - Process Equipment
    - Effluent/Exhaust System
Overview

- What are HPM Liquids and Solids?
  - **Ampoules**
    - Typically low quantities of “exotic” compounds used for metal deposition and coating
  - What forms do they come in?
    - Liquids
    - Vapor from liquids and solids
Overview

- What are ampoules or bubblers and what do they do?
  - Design
Overview

- What are the Systems that use ampoules and what do they do?

  - Elements
    - Enclosure
    - Canister /Cylinder
    - Connections, Valving and Piping
    - Process Equipment
    - Effluent/Exhaust System

  - System may also include:
    - Heater
    - Evaporator
    - Process pump
Steps to installation and permitting of an HPM Liquid of Solid chemistry system

1. Hazard Assessment
2. Rank Hazards and Determine Fault Tolerance
3. Define Controls
4. Review Requirements
5. Approach Permitting Agency
1. Hazard Assessment
   A. Assess the chemical hazards first
      - MSDS review
      - Internet search
      - Discussions with manufacturer
   B. Understand the delivery system
      - Design review
      - PHA
      - Discussions with vendors and designers

Hint: call ahead to find out lead times and process for Fire Department Permit
2. Rank Risks and Determine Fault Tolerance
   A. Ranking
      ‣ Understand foreseeable failures
      ‣ Predict the severity of a failure(s)
      ‣ Determine the likelihood for that failure
   B. Fault Tolerance
      ‣ No single points of failure
      ‣ If severity is assessed to be debilitating injury or death, use three-points of failure
3. Define Controls

- Use engineering controls for primary controls
  - Rely on administrative controls as back-ups
- Examples:

<table>
<thead>
<tr>
<th>Failure</th>
<th>Control Options</th>
</tr>
</thead>
<tbody>
<tr>
<td>Overflow</td>
<td>Level sensor, spill detection, secondary containment, drains</td>
</tr>
<tr>
<td>Leak out of system</td>
<td>Leak sensors, spill detection, secondary containment</td>
</tr>
<tr>
<td>System overheating</td>
<td>Thermal sensors, over-current protection</td>
</tr>
<tr>
<td>System over pressure</td>
<td>Pressure sensors, pressure relief devices</td>
</tr>
</tbody>
</table>
Review Requirements

Check the codes
- California Fire Code
- California Building Code
- Locally Required Fire Codes
- Local Building Code
- CalARP

Regulations that may need attention
- BAAQMD
- Water Permits
- Hazardous Waste
- CFATS
- Cal-OSHA
HPM Liquids and Solids

- Apply for Fire Department Permit
  - Inspector may want to do a plan check first
    - Usually requires some form of drawings
    - Inventory of chemicals with Safety Data Sheets
    - Equipment data sheets
  - A plan check meeting may be required
    - Some jurisdictions want to meet with EHS and Facilities team to discuss project and understand issues
      - Architects and Construction Project Managers may be needed
- Prep for a site walk
  - Address all plan check comments
Case Study 1:
- Install 55 gallon TMAH (tetramethylammonium hydroxide) bulk delivery system
  - Needed for a photolithography wafer strip wet bench
  - Wet bench will have two wafer strip baths and two DI Water rinse baths
I. TMAH Hazard Assessment
   A. Assess the chemical hazards first
      ▶ MSDS review
      ▶ Ammonia exposure and odors are a major concern
   B. Understand the delivery system
      ▶ Reviewed and approved Bulk Chemical Delivery System
      ▶ Reviewed and approved Wet Bench
      ▶ Reviewed and approved Acid Waste Neutralization system
      ▶ In-house Project Manager is using local vendor for install and plumbing
2. **Rank Risks and Determine Fault Tolerance**

A. **Risks**
   - Drum mishandling
   - Improper connection
   - Leaks in BCDS
   - Improper plumbing
   - Leaks at wet bench
   - Odors and vapors from bench
   - Leaks at AWN
   - AWN malfunction

   Which ones need two controls?
### Define Controls

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<tr>
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<th>Control Options</th>
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<tbody>
<tr>
<td>Improper connection, improper plumbing</td>
<td>Leak check before commissioning</td>
</tr>
<tr>
<td>Leaks in BCDS, leaks at wet bench, odors and vapors from bench, leaks at AWN</td>
<td>Ammonia sensors, spill detection, secondary containment</td>
</tr>
<tr>
<td>AWN Malfunction</td>
<td>Failure alarms and notification</td>
</tr>
</tbody>
</table>
Review Requirements

Check the codes
- Chapter 27 - Semiconductor
- Chapter 50 - Hazardous Materials
- Chapter 54 – Corrosive Materials
- Chapter 60- Highly Toxic and Toxic Materials

Regulations that may need attention
- Water Permit for the AWN
- Hazardous Waste – will affect the hazardous waste permit, will make you a LQG in California
Agenda

Material Considerations

ESH Aspects of III-V Processing
  Chemical delivery
  Process by-products
Material Considerations
Drivers for New Material Development

- Lower thermal budgets
- Controlling reactivity in ALD
- Non-PFC etch replacements
- Chamber cleans of ‘resistant’ materials
- Higher material utilization
- Mobility enhancement

ESH Concerns:
- Toxicity
- Reactivity
Organometallic – Chemical and Physical Hazards

- Most Metal organics (MO) sources are clear, colorless liquids/white solids
- Water reactive
  - Highly reactive towards water to form hydroxides
- Pyrophoric
  - Readily oxidized in air to form solid metal oxides
- Stable in inert gases, such as argon and nitrogen
- MO leaks present a significant hazard
  - Fire and potential for explosion
  - Contamination of equipment and facilities
  - Planning and design for source controls and containment is critical
ESH Aspects Processing with Energetics

• Material Characterization
• On-site Transport, Storage and Handling
  – Materials Management
• Delivery Systems
  – Design Criteria – on board, remote, basic designs
  – System Controls – protective systems
  – Material Properties

• Process Equipment
  – Reactive Chemistry
  – PM Criteria

• Support Systems / Abatement
  – Design options / Design for Maintenance
  – By-product Characterization / Identification
  – Foreline / Duct / Abatement Cleaning
  – Hazard Controls – protective structures

• Waste Disposal / Parts Handling
  – Safety Protocols
  – Shipping Criteria - Classifications
On Board Metal Alkyls - Chemical Reaction Potential

- Metal Alkyls are withdrawn primarily by two methods when using the vapor phase
  - Sub atmospheric withdrawal requires the product to be sucked out using a vacuum
  - Carrier gas that is bubbled through the liquid/solid and becomes the saturated with the vapor (Bubbler)

- To maintain a constant concentration, the bubblers maybe heated in a bath to a fixed temperature

- Of concern is the common practice of using a liquid heating fluid such as glycol
  - Some of the metal alkyls are violently water reactive
    » Glycols behave in a manner similar to water
Trimethyl Aluminum Fire

- Testing has shown that a considerable amount of liquid Organometallics can be released through the VCR leak check hole.

- Testing has determined
  - A VCR leakcheck hole 2.38 mm dia. with a system pressure of 0.7 bar (10 psig) results in Trimethyl Aluminum flowing out 10.7 gms/sec
    - In open air this sprayed a distance of 170 cm (5.5 feet). This atomized the liquid TMAI creating an intense flame
    - Note - Very little liquid reached the ground (10%)

Source: Eugene Ngai, Chemically Speaking, LLC
Trimethyl Aluminum (TMAI) was being fed from a bulk system to an on board host container.

A VCR connection was not leak checked and was loose. Liquid TMAI shot out of the connection causing a fire which was detected by the UV/IR Sensor.

The UVIR sensor shutdown the system; with pressure removed from the line, the connection developed a crust of aluminum oxide and plugged.

Fire was contained in the gas box causing minimal damage.

Created a considerable amount of aluminum carbide particles.
What’s Happening After the Process?

- Depends on the process
  - Co-reactants
  - Non stoichiometric combustion: may be delayed events associated with non-volatile coatings developing surfaces
  - Catalytic effects of surfaces
  - Solid by-products of combustion
  - Pressure
  - Recipe Times

Source: Matheson
The Problem with By-Products

The survey says......

Source: SEMATECH

Number of Events

Energetic Event Location

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Disilane Event

- Controlled introduction of air in a balance with N₂
  Stepwise and tightly controlled
- Destroyed flex bellows and KF clamps
- Two inch steel exhaust bent 180°
- Steel fragments thrown to 10 meters
- Steel panels on the abatement unit (Guardian) knocked off
- Black sooty stains found on the remaining sections

Source: Micron
## Reaction Compounds

<table>
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<tr>
<th>Layers Processed</th>
<th>Etchants Considered</th>
<th>Possible R-B compounds</th>
</tr>
</thead>
<tbody>
<tr>
<td>GaAs</td>
<td>HF</td>
<td>AsH&lt;sub&gt;3&lt;/sub&gt;</td>
</tr>
<tr>
<td>GaN</td>
<td>HCl</td>
<td>As&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;3&lt;/sub&gt;</td>
</tr>
<tr>
<td>InP</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;2&lt;/sub&gt;</td>
<td>AsCl&lt;sub&gt;3&lt;/sub&gt;</td>
</tr>
<tr>
<td>InGaAs</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O</td>
<td>AsF&lt;sub&gt;3&lt;/sub&gt;</td>
</tr>
<tr>
<td>AlGaAs</td>
<td>Other</td>
<td>PH&lt;sub&gt;3&lt;/sub&gt;</td>
</tr>
<tr>
<td>InAlAs</td>
<td></td>
<td>P&lt;sub&gt;4&lt;/sub&gt;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>InCl&lt;sub&gt;3&lt;/sub&gt;</td>
</tr>
<tr>
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</tr>
<tr>
<td></td>
<td></td>
<td>GaF&lt;sub&gt;3&lt;/sub&gt;</td>
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Fire Safety
Fire Detection for Energetics

- Energetic Materials
  - Pyrophoric Liquids
  - Class III Unstable Water Reactives
    - TMA, TMI, TMG, DEZ, TSA,...
- IFC 27.2.3.4 (6) & NFPA 318 11.2.6.1
  - Provide UV/IR, HSSD, or “other” approved detection
- Sematech BKM Bulk Delivery [10.4.5]
  - HSSD and
  - Flame Detection (or redundant HSSD)
- Sematech BKM Gas Box or Ampule Delivery [11.2.5]
  - HSSD and
  - Flame Detection (or redundant HSSD)
# IFC & NFPA (318)
## Maximum Quantities of HPM @ Workstation

<table>
<thead>
<tr>
<th>HPM CLASSIFICATION</th>
<th>STATE</th>
<th>IFC TABLE 1805.2.2 MAXIMUM QUANTITY</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flammable</td>
<td>Liquid</td>
<td>15 gallons a, b</td>
</tr>
<tr>
<td></td>
<td>Solid</td>
<td>5 pounds a, b</td>
</tr>
<tr>
<td>Corrosive</td>
<td>Gas</td>
<td>150L Combined Open : 25 gallons b</td>
</tr>
<tr>
<td></td>
<td>Liquid</td>
<td>Closed : 150 gallons b, e</td>
</tr>
<tr>
<td></td>
<td>Solid</td>
<td>20 pounds a, b</td>
</tr>
<tr>
<td>Highly toxic</td>
<td>Liquid</td>
<td>15 gallons a</td>
</tr>
<tr>
<td></td>
<td>Solid</td>
<td>5 pounds a</td>
</tr>
<tr>
<td>Oxidizer</td>
<td>Gas</td>
<td>150L Combined Open : 12 gallons b</td>
</tr>
<tr>
<td></td>
<td>Liquid</td>
<td>Closed : 60 gallons b</td>
</tr>
<tr>
<td></td>
<td>Solid</td>
<td>20 pounds a, b</td>
</tr>
<tr>
<td>Pyrophoric</td>
<td>Liquid</td>
<td>0.5 gallon c, f</td>
</tr>
<tr>
<td></td>
<td>Solid</td>
<td>See Table 1804.2.2.1</td>
</tr>
<tr>
<td>Toxic</td>
<td>Liquid</td>
<td>Open : 15 gallons b</td>
</tr>
<tr>
<td></td>
<td>Solid</td>
<td>Closed : 60 gallons b</td>
</tr>
<tr>
<td></td>
<td></td>
<td>5 pounds a, b</td>
</tr>
<tr>
<td>Unstable reactive Class 3</td>
<td>Liquid</td>
<td>0.5 gallon a, b</td>
</tr>
<tr>
<td></td>
<td>Solid</td>
<td>5 pounds a, b</td>
</tr>
<tr>
<td>Water-reactive Class 3</td>
<td>Liquid</td>
<td>0.5 gallon c, f</td>
</tr>
<tr>
<td></td>
<td>Solid</td>
<td>See Table 1804.2.2.1</td>
</tr>
</tbody>
</table>

- a. Quantities shall be increased 100 percent for closed system operations. Note a & b can be combined.
- b. Quantities shall be increased 100 percent when workstation protected with approved FSS. Note a & b can be combined. Note b & e can not combine.
- c. Allowed only in workstations that are internally protected with an approved FSS and compatible with the reactivity of materials in use at the workstation.
- d. The quantity limits apply only to materials classified as HPM.
- e. Quantities shall be increased 100 percent for nonflammable, noncombustible corrosive liquids when the materials of construction for workstations are listed or approved for use without approved FSS. Note b & e can not combine.
- f. A maximum quantity of 5.3 gallons shall be allowed at a workstation when conditions are in accordance with Section 1805.2.3.4.
Fire Suppression

• Fire Safety of Energetic Materials Introduces Many New Challenges
• Suppression doesn’t currently exist!
  • *Most Energetics are Incompatible with Water*
  • *Fire Sprinklers Are Incompatible*
• Gaseous Suppression “Band-Aid”
  • Exhaust is Required for Process Safety
  • Pyrophoric Re-Ignites after Agent Exhausted
• Purging & Smoke Control Considered
  • Nitrogen Purge After Fire Detection (Not “Listed” N2 FSS)
  • Increase Exhaust Upon Fire Detection
    • To Capture Smoke Particles
    • Smoke Development vs. Capture Velocity Unknown
• Class D Dry Powder for Metal Fires
  • Portable Extinguishers Only (No Auto)
• **Current ➔ Early Detection Minimize Fuel Load**
  • Challenges with Proprietary Chemicals & Actual Fire Testing
  • What are you doing with Fire Alarm Signals?
• **Future Options ➔ To Be Developed?**
  • KFPI is conducting TMA testing for Suppression Options
  • October 2015!!
Fire Detection Hazards

- Different Fire Hazards @ Different Points in the Process
- Different Fire Hazard @ SAME location in Process
  - Upstream or Downstream of Bubbler
    - Upstream 100% Energetic
    - Downstream 1000:1 H2

<table>
<thead>
<tr>
<th>Fire Risk</th>
<th>Fire Classification</th>
<th>Flame Characteristic</th>
<th>HSSD Characteristic</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bulk Supply</td>
<td>Energetic</td>
<td>Energetic</td>
<td>Energetic</td>
</tr>
<tr>
<td>Gas Box Before Bubbler</td>
<td>Energetic</td>
<td>Energetic</td>
<td>Energetic</td>
</tr>
<tr>
<td>Gas Box After Bubbler</td>
<td>Hydrogen</td>
<td>Hydrogen</td>
<td>Hydrogen</td>
</tr>
</tbody>
</table>
Fire Detection Application

• Quiz *(Real Tool in Fab Example)*
• Find this Detector in your Tool Gas Box...
  • TMA, TMI, TMGa

Flame Detector Won’t Detect Hydrogen Fire
*“Hope the HSSD Works!”*

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</thead>
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<tr>
<td>Gas Box Upstream</td>
<td>Energetic</td>
<td>Energetic</td>
<td>Energetic</td>
</tr>
<tr>
<td>Gas Box Downstream</td>
<td>Hydrogen</td>
<td>Hydrogen</td>
<td>Hydrogen</td>
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</tbody>
</table>

Table 1: Response Testing

<table>
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<tr>
<th>Fuel</th>
<th>Size</th>
<th>Distance (ft/m)</th>
<th>Average Response Time (Seconds)</th>
</tr>
</thead>
<tbody>
<tr>
<td>n-Heptane</td>
<td>1' x 1'</td>
<td>150/45.7</td>
<td>8.5</td>
</tr>
<tr>
<td>Methanol</td>
<td>1' x 1'</td>
<td>40/12.2</td>
<td>5.36</td>
</tr>
<tr>
<td>Methane</td>
<td>36&quot; Phume</td>
<td>120/36.6</td>
<td>3.1</td>
</tr>
<tr>
<td></td>
<td>16&quot; Phume</td>
<td>60/18.3</td>
<td>4.0</td>
</tr>
<tr>
<td>Propane</td>
<td>1' x 1'</td>
<td>90/27.4</td>
<td>2.2</td>
</tr>
<tr>
<td>Jet Fuel</td>
<td>1' x 1'</td>
<td>90/27.4</td>
<td>3.9</td>
</tr>
<tr>
<td>Diesel</td>
<td>1' x 1'</td>
<td>50/15.2</td>
<td>2.1</td>
</tr>
<tr>
<td>Lube Oil</td>
<td>1' x 1'</td>
<td>60/18.3</td>
<td>2.0</td>
</tr>
<tr>
<td>Ethanol</td>
<td>1' x 1'</td>
<td>120/36.6</td>
<td>2.8</td>
</tr>
<tr>
<td>Gasoline</td>
<td>1' x 1'</td>
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HSSD for Energetics

- Most Energetics “Believed” to have Large Smoke Release
- Airflow Increases Dilution
- Dilution Lowers Concentration
- Exhaust is Not Recirculating
- Smoke % Concentration is VERY LOW
- HSSD Factory Defaults ➔ NOT EFFECTIVE
- SPOT Smoke Detectors ➔ NOT EFFECTIVE

<table>
<thead>
<tr>
<th>Signal</th>
<th>Concentration</th>
<th>Time Delay</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alert</td>
<td>0.025% obs/ft</td>
<td>10sec</td>
</tr>
<tr>
<td>Action</td>
<td>0.044% obs/ft</td>
<td>10sec</td>
</tr>
<tr>
<td>Fire 1</td>
<td>0.063% obs/ft</td>
<td>10sec</td>
</tr>
<tr>
<td>Fire 2</td>
<td>0.625% obs/ft</td>
<td>10sec</td>
</tr>
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</table>
Real Smoke Event

- Upgrade Installation → Crossed Wires
- Thermal Event → No Exhaust Ventilation
- Highest Data = 0.026% obs/ft.
- Missed Factory Defaults
  - Alert Relay Not on “mini-units”
- 1st & 2nd Alarms by KFPI setup
- Exhaust → Lower Smoke % from Event

Wire Burn Data
Same Source Different Compartments

<table>
<thead>
<tr>
<th>Test #</th>
<th>Location</th>
<th>Max Smoke % obs/ft</th>
<th>Duration</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>PM (Off)</td>
<td>0.1428</td>
<td>1:32</td>
</tr>
<tr>
<td>17</td>
<td>HV</td>
<td>0.0072</td>
<td>0:27</td>
</tr>
<tr>
<td>19</td>
<td>HV</td>
<td>0.0109</td>
<td>0:13</td>
</tr>
<tr>
<td>20</td>
<td>PM (On)</td>
<td>0.0428</td>
<td>1:07</td>
</tr>
<tr>
<td>21</td>
<td>FPSU (TSFR)</td>
<td>0.0653</td>
<td>1:50</td>
</tr>
<tr>
<td>22</td>
<td>FPSU</td>
<td>0.0194</td>
<td>0:26</td>
</tr>
<tr>
<td>23B</td>
<td>FPSU</td>
<td>0.0281</td>
<td>0:39</td>
</tr>
<tr>
<td>27</td>
<td>CR</td>
<td>0.0231</td>
<td>1:39</td>
</tr>
<tr>
<td>28</td>
<td>SCCT</td>
<td>0.0459</td>
<td>0:49</td>
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Signal | Concentration  | Time Delay |
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Fire Detection Application

- Back to our Quiz
- Find this Detector in your Tool Gas Box...
  - TMA, TMI, TMGa
- Flame Detector Won’t Detect Hydrogen Fire
- Unlike Pyrophoric, **Hydrogen Fire → ~No Smoke**

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"How Do We Protect the Hydrogen Fire Risk?"

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<tbody>
<tr>
<td>Gas Box Upstream</td>
<td>Energetic</td>
<td>Energetic</td>
<td>Energetic</td>
</tr>
<tr>
<td>Gas Box Downstream [1000:1 H2]</td>
<td>Hydrogen</td>
<td>Hydrogen</td>
<td>Hydrogen (Ineffective)</td>
</tr>
</tbody>
</table>
How to Protect the Risk

• Flame Detectors MUST be Approved for Hazards:
  • Hydrocarbon Fires
  • Silane Fires
  • Hydrogen Fires

• HSSD MUST be used:
  • Alarm % Concentration Settings MUST be:
    • As Low As Possible to be Reliable
    • Not Too Low for False Alarms

• Important to have ERT Plan for All Alarm Signals, Types, per Chemical Hazard.

• Fire Suppression Methods Still Be Tested & Evaluated
  • Fire Sprinklers Should NOT be used

Make Sure Your Fire Safety is NOT Just a Decoration