

## CMC Seminar 2019

### Precursors and Specialty Gases: Greater Needs and Challenges on Characterization and Quality Control

Berry TSENG, Product Quality Director  
17 Oct. 2019

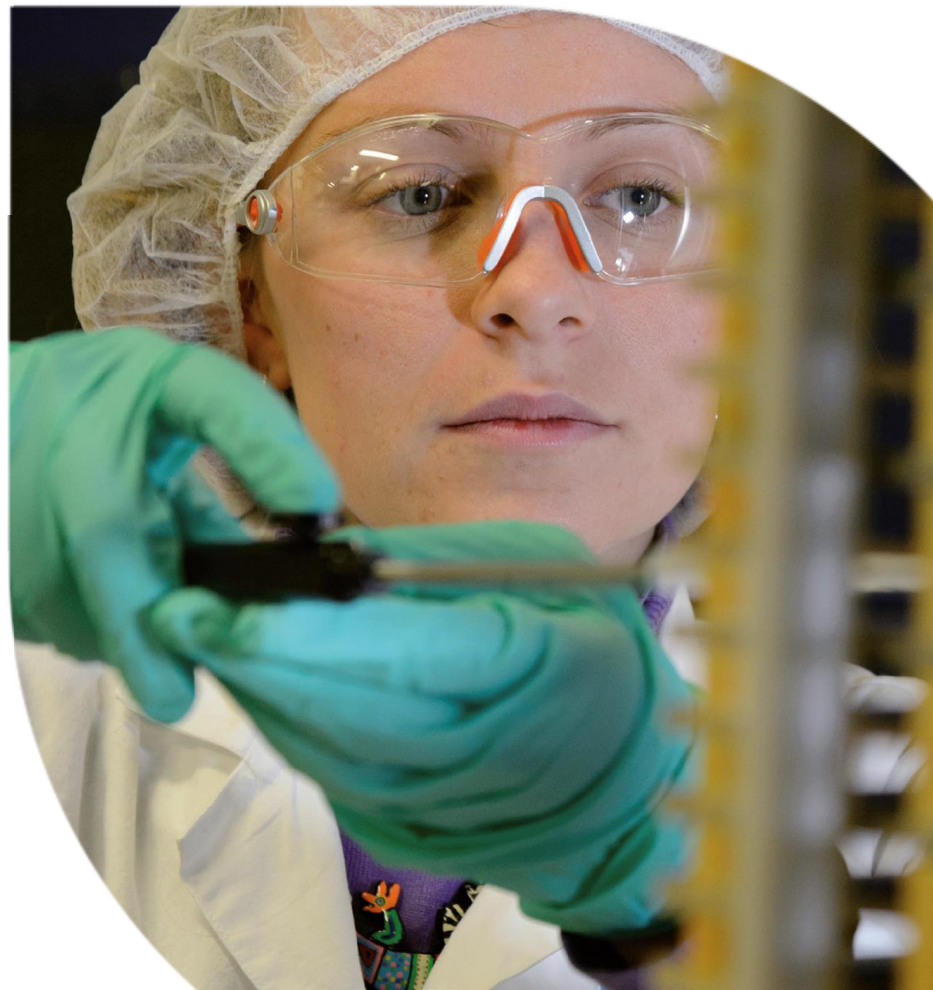
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# Major trends and challenges are shaping our materials

Growing **Novel Material** in flux

- **ALD/CVD precursors for advanced node development**
- New material learning cycle impact to ramp/HVM
- Classical metrology is inadequate to tell good/NG
- Product “in spec” is not necessarily “fit for use”

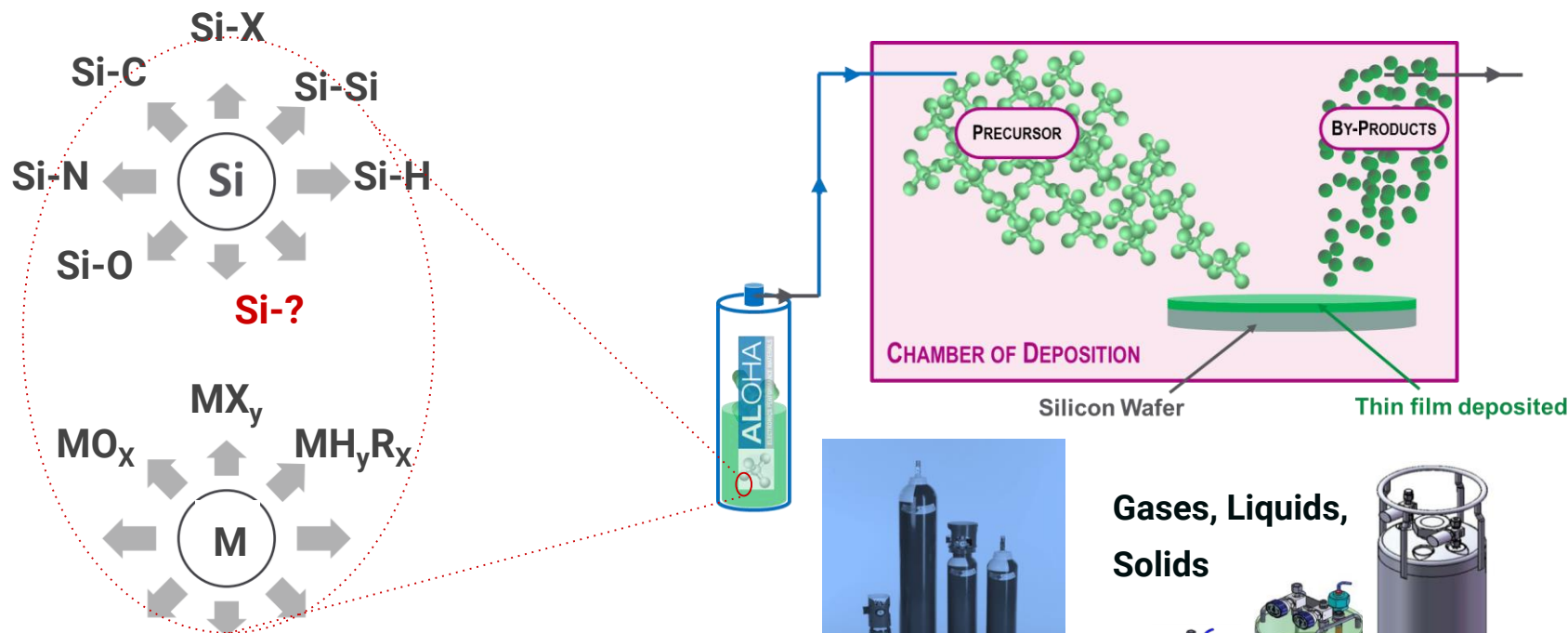
**Zero Excursion -**  
Lowest Defect Density

- **Unstable/sensitive material characterization** from POS to POU
- Killer defect detection vs. vast amount of nuisances
- Inline/offline correlation
- Increasing unwanted impurities

**Product consistency and process stability**

- **Dynamic control limit tightened** from the scope of material to run-to-run wafer
- Fast QC verification and real-time feedback for endpoint control, fault detection and drift control
- Baseline shift early detection and deviation control

# Precursor chemistries and difficulties



**M: Al, Zr, Hf, Ti, Co, Mo, W, Ru...**

**L : multi ligand options**

# Precursor characterization challenge

- ❑ **Highly reactive**
- ❑ **Complex matrix**
- ❑ **Scarce information**

Analytical techniques and methods must be carefully selected and thoroughly studied.

Instrument	Applications
ICP-MS	trace metals
GC/MS	assay, impurity identification
IC	Anions (Fluoride, Chloride etc)
FTIR	functional group ID, impurity quantification
UV-Vis	impurity quantification
LC	impurity quantification
Karl Fischer	Water content
TGA	Thermal stability, weigh loss



Sophisticated techniques such as GC/MS, NMR, ICP-MS and FT-IR can provide essential information about trace-level contaminants

# Precursor characterization challenge (cont./)

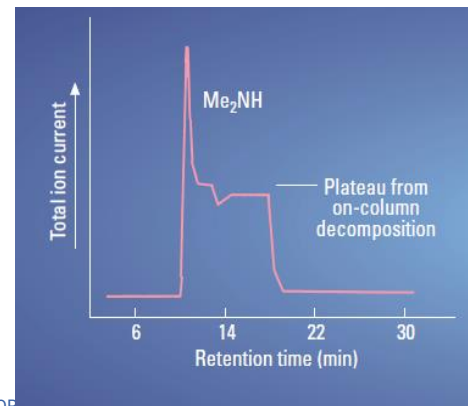
**The reactive nature of the precursors is the major challenge for sample preparation and assay analysis**

## Sample handling/Preparation:

- Glove box is required for sample handling
- Great care needed to prevent sample from decomposing: dry solvents, dry sample vials etc.

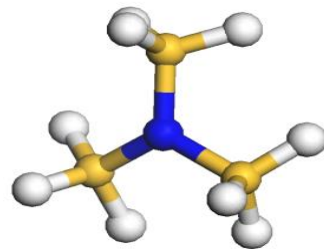
## Instrumentation difficulties:

- Syringe clog
- Strong interact with GC column stationary phase damages the column
- Corrosive or non-volatile residues contaminate/destroy detectors: SiO<sub>2</sub> coating, HCl Corrosion
- Frequent maintenance required to ensure instrument functionality



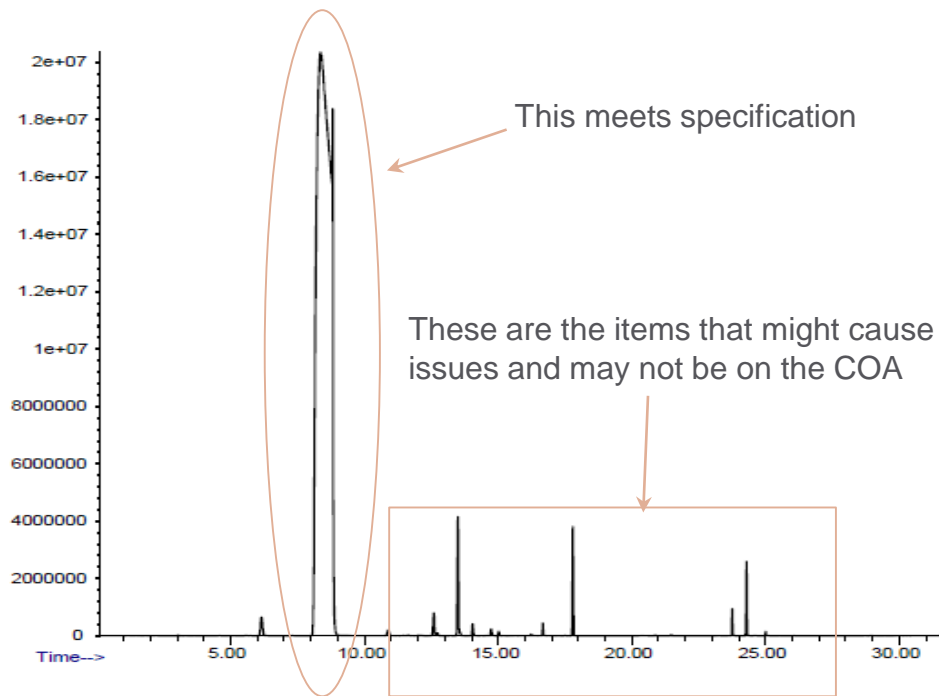
# Precursor quality control plan - greater needs

- **Starting Material + Compound(s) = Final Material (Purification/Synthesis)**
  - “Fingerprint (beyond CoA)” final material and critical starting material
  - Active batch variation to validate spec limits for CoA parameters
  - Complete baseline to comprehend variability
- **Final Material + Container(s) = Intermediate/Final Product(s)**
  - Packaging is as important as chemical itself
  - decon/clean, integrity leak/moisture check
- **Final Product + Process = Deposition**
  - Co-process optimization to validate material in use consistency





# Specialty gases characterization challenge



- What happened?

- Decomposed over time (ex. B2H6 - > B4H10)
- Product rearrangement over time (ex. DCS -> MCS, TCS)
- Unknown peaks, phase change impurities...etc

- What to do?

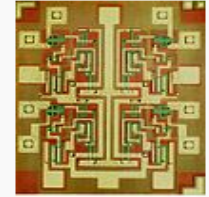
- Revisit unstable, sensitive SG
- Obtain data over the shelf life
- Understand and control “material” and “material in use”
- Make a “correlation” with process



# Specialty gases characterization challenge (cont./)

- Detect and eliminate **trace metallic particles (killer defect)** in critical etch materials
  - New flavor of ICP-MS: GED-sp-ICP-MS application under developing
  - Nanoparticle extraction and characterization by ICP-MS complements traditional particle technique by providing additional info of elemental composition plus particle size/concentration
- Characterize **unwanted decomposed byproducts** in critical materials (ex. B<sub>2</sub>H<sub>6</sub>)
  - Traditional FTIR to semi-quantitatively determine higher boranes
  - Inline sensor complements critical material in use (ex. hydrides)

## Semiconductor manufacturing processes



10  $\mu\text{m}$  – 1971  
6  $\mu\text{m}$  – 1974  
3  $\mu\text{m}$  – 1977  
1.5  $\mu\text{m}$  – 1982  
1  $\mu\text{m}$  – 1985  
800 nm – 1989  
600 nm – 1994  
350 nm – 1995  
250 nm – 1997  
180 nm – 1999  
130 nm – 2001  
90 nm – 2004  
65 nm – 2006  
45 nm – 2008  
32 nm – 2010  
22 nm – 2012  
14 nm – 2014  
10 nm – 2017  
7 nm – ~2019  
5 nm – ~2021



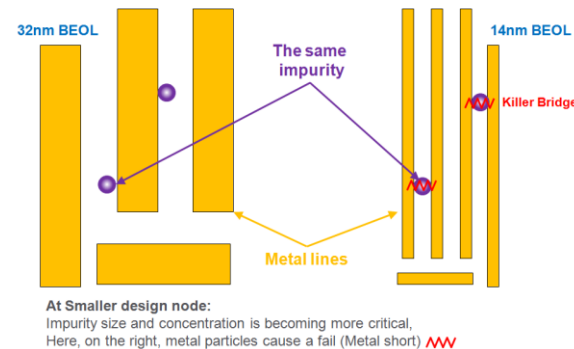
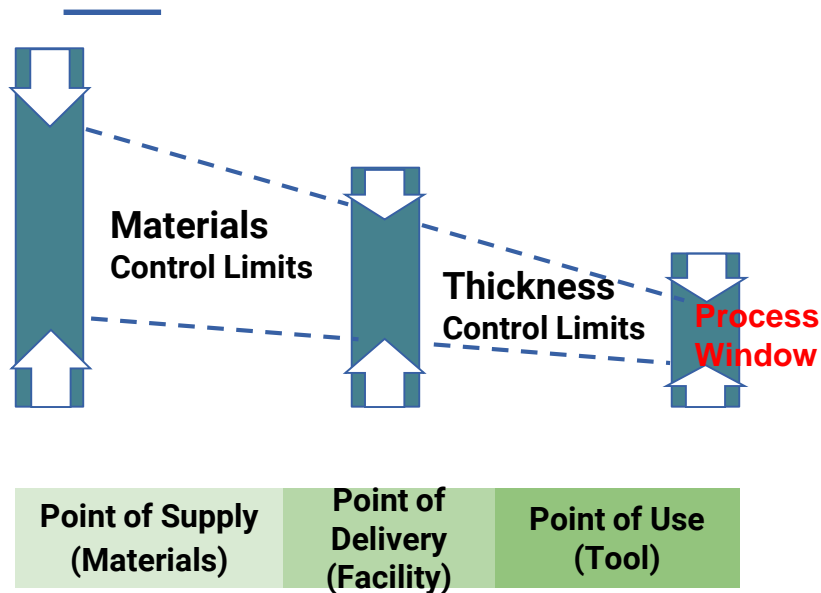
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# Quality control requirements - yesterday vs. today

Yesterday	Today
<ul style="list-style-type: none"><li>✓ “In Spec” product (STS)</li><li>✓ Historical quality data review (SQC)</li><li>✓ Few key parameters</li><li>✓ Metrology calibrated</li></ul>	<ul style="list-style-type: none"><li>✓ “<u>In Control</u>” product (STC)</li><li>✓ Real-time process control wi/ OCAP(SPC)</li><li>✓ Dynamic key parameters</li></ul>
<ul style="list-style-type: none"><li>✓ Just in Time Supply (JIT)</li><li>✓ Casual PCN at major change</li><li>✓ Raw material non conformity management</li><li>✓ CofC or CofA (paper format)</li></ul>	<ul style="list-style-type: none"><li>✓ Process capability analysis (IDL, gage R&amp;R, LTS)</li><li>✓ Business Continuity Plan (BCP)</li><li>✓ “Copy Exact” (Management of Change from R&amp;D to HVM)</li><li>✓ CoA reliance/upstream supplier management/lot traceability</li><li>✓ CofA (e-CoA pre-upload review)</li></ul>

**Tomorrow?**

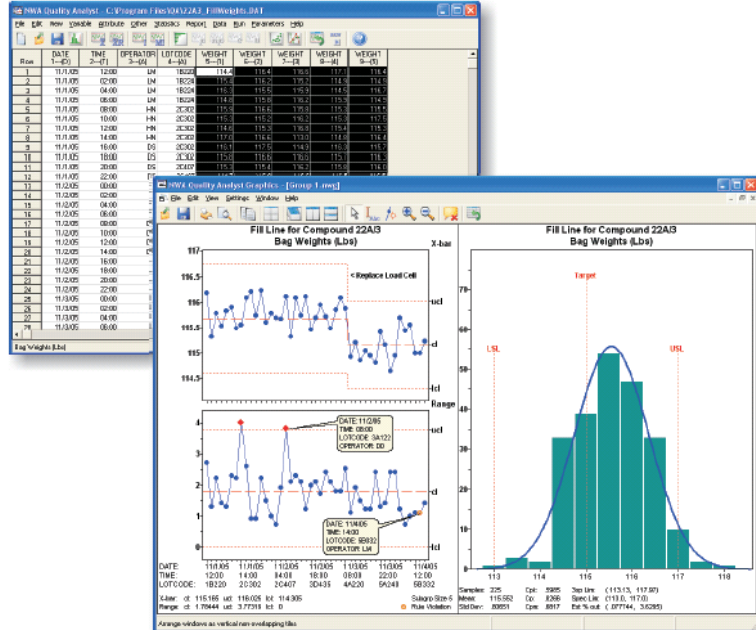
# Shrinking of process window leading to stringent QC



- As customer process window is shrinking to smaller nodes and zero defect, “materials window” requires tighter acceptance limits adjusted dynamically with higher frequency than in the past.
- “Co-process control” to beat down process variability.

# Quality control requirement - tomorrow?

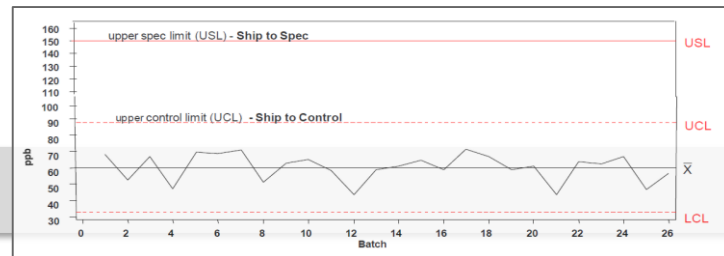
- Process parameter trending in CoA
- Production-like pre-conditioning
- Enhanced sub-supplier defence line
- Baseline management (technology, application, factory-specific)
- Selective for cost-effective quality
- Predictive data analytics - advanced process control by real-time SPC + correlation (from corrective to predictive)
- .....



# QMS focusing on entire supply chain control

**Quality Incident Free** can only be achieved in a sustainable manner by exercising proper control on products and processes and cultivating quality mindset over the entire supply chain.

Over the entire material supply chain



**Supplier  
Quality  
Management**

**Process Control System - SPC, STC, Baseline Management**

**Validation of  
specifications**

**Metrology  
Control**

**Material  
Deviation  
Review**

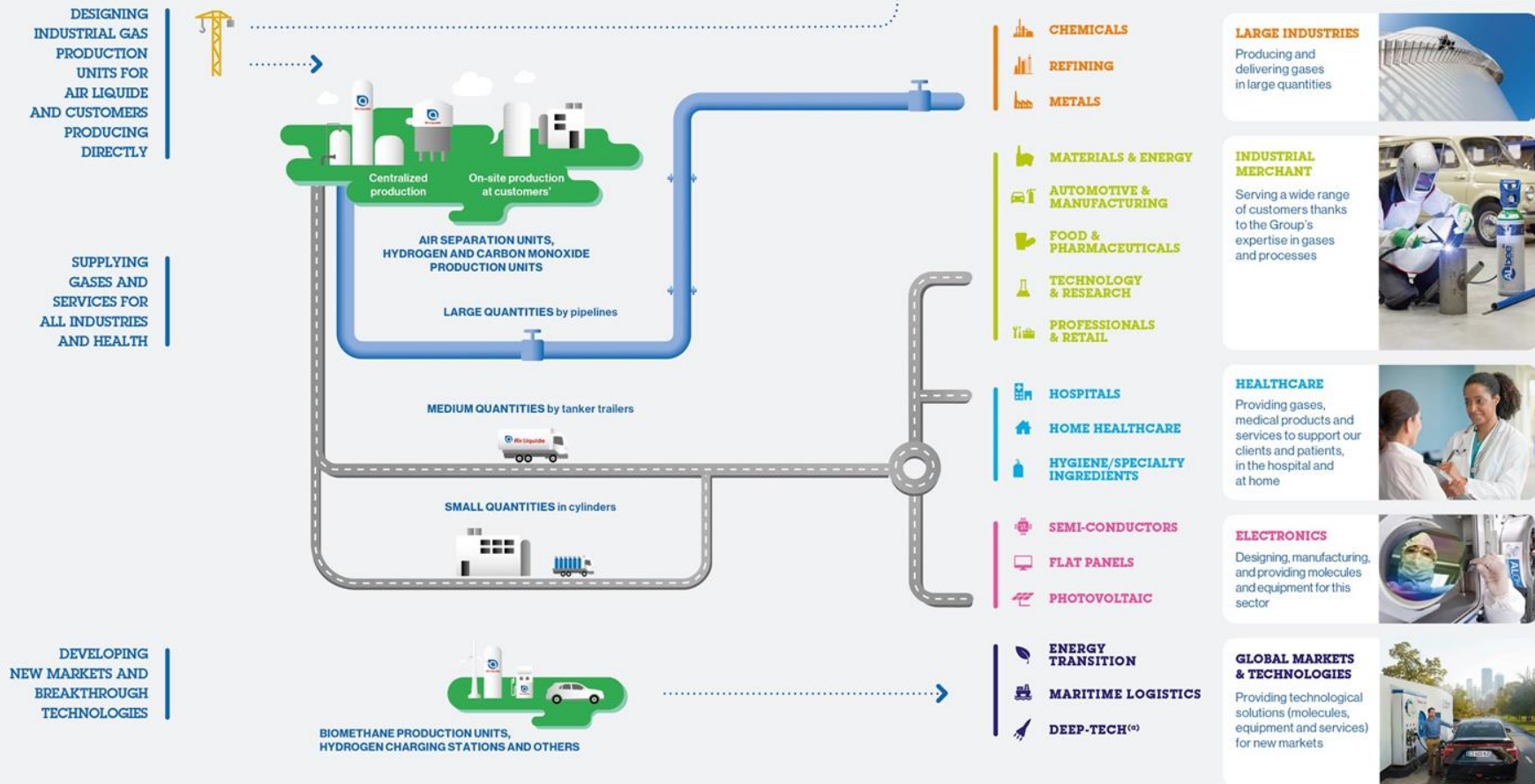
**Change  
Management**



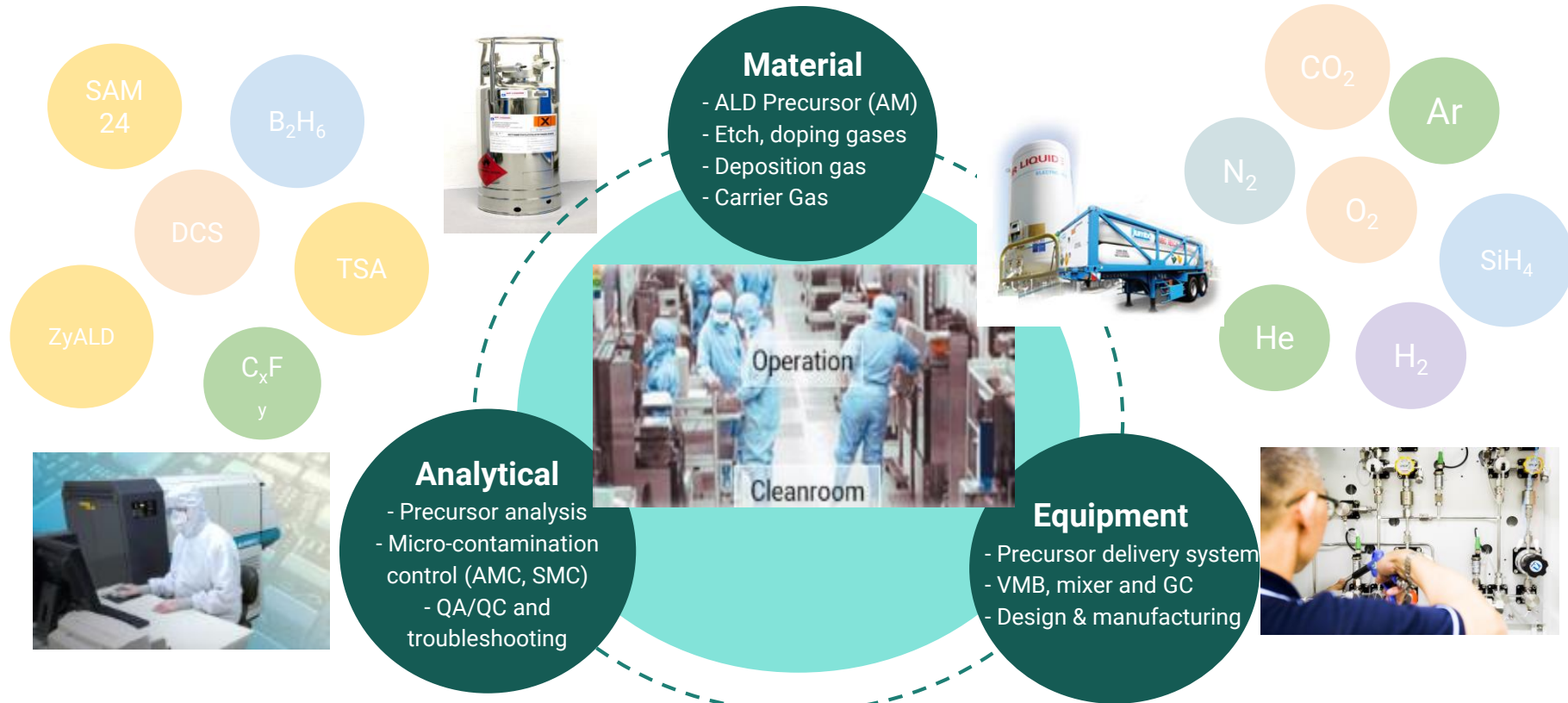
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# Air Liquide global activities



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# Thank You ! Q&A

Contact [berry.tseng@airliquide.com](mailto:berry.tseng@airliquide.com) for more information.

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