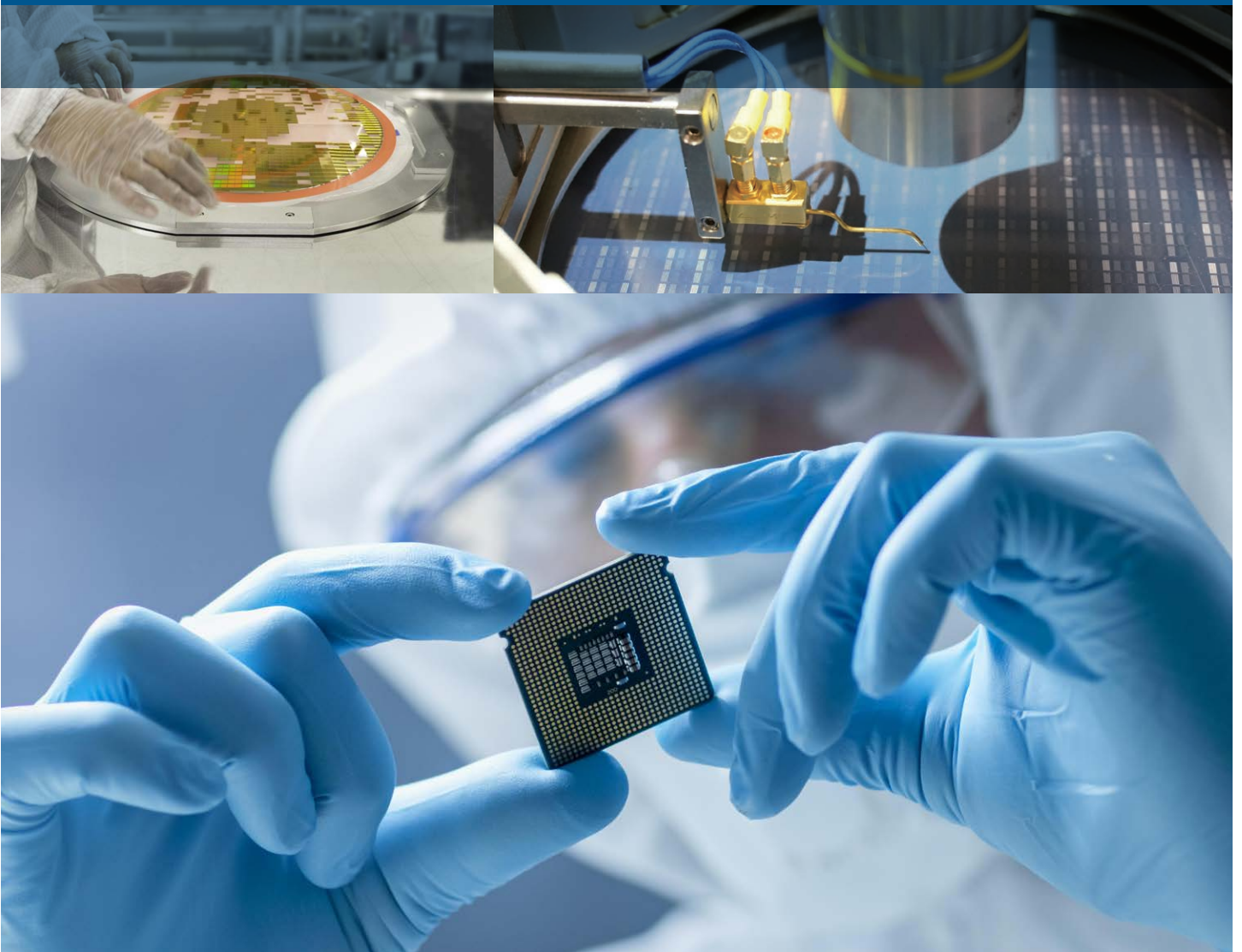
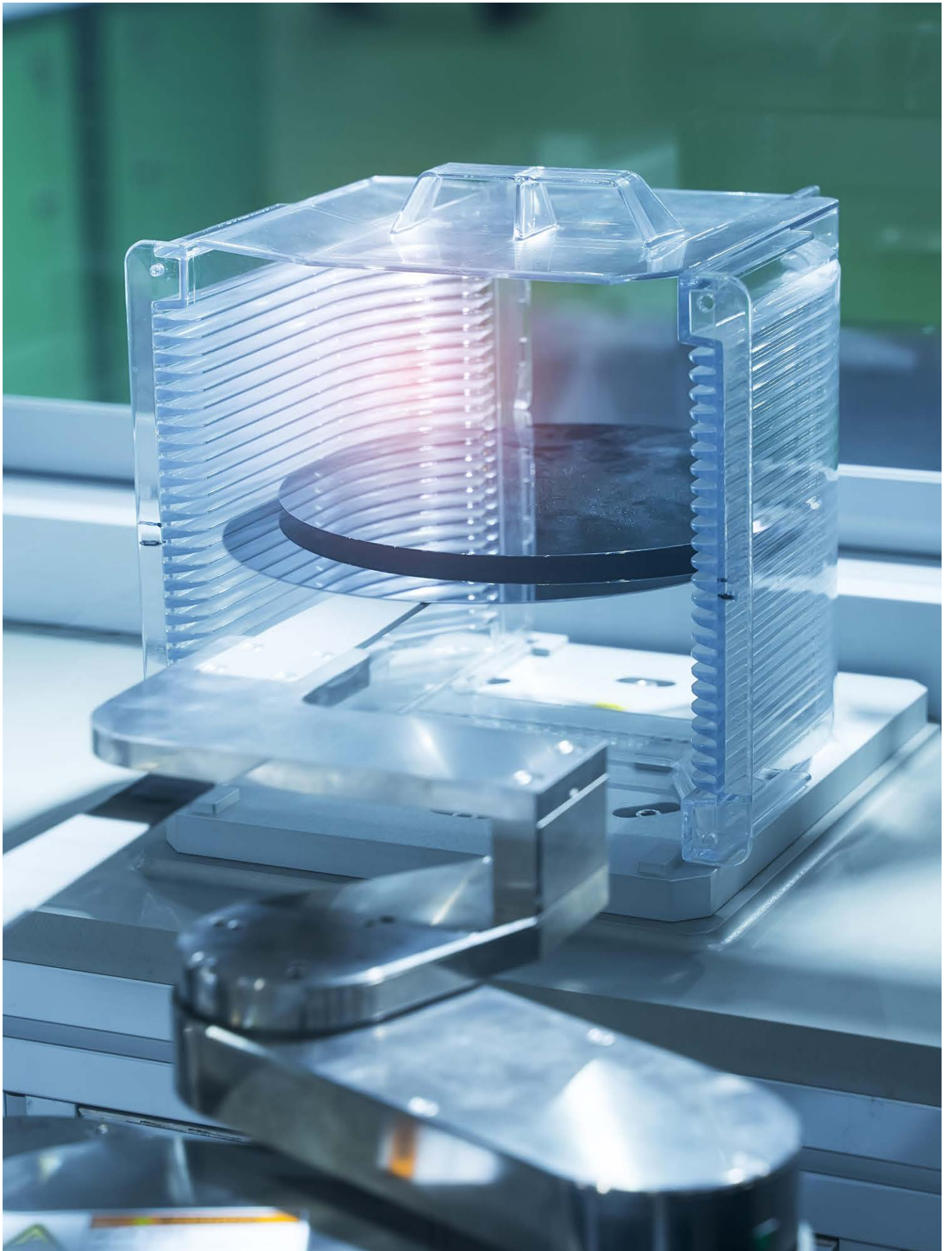


# Agilent Semiconductor Industry Solutions





# Introduction

Semiconductors are the core of modern electronic products and are used in smart phones, cars and other fields. With advances in the semiconductor industry, many devices have become smaller, faster, more reliable, and more powerful.

In order to solve the problem of high cost, product yield during semiconductor manufacturing has attracted the most attention from semiconductor manufacturers. There are many factors that affect product yield, of which contamination is undoubtedly one of the most important factors. Industry experts have estimated that contamination causes around 50% of production loss.

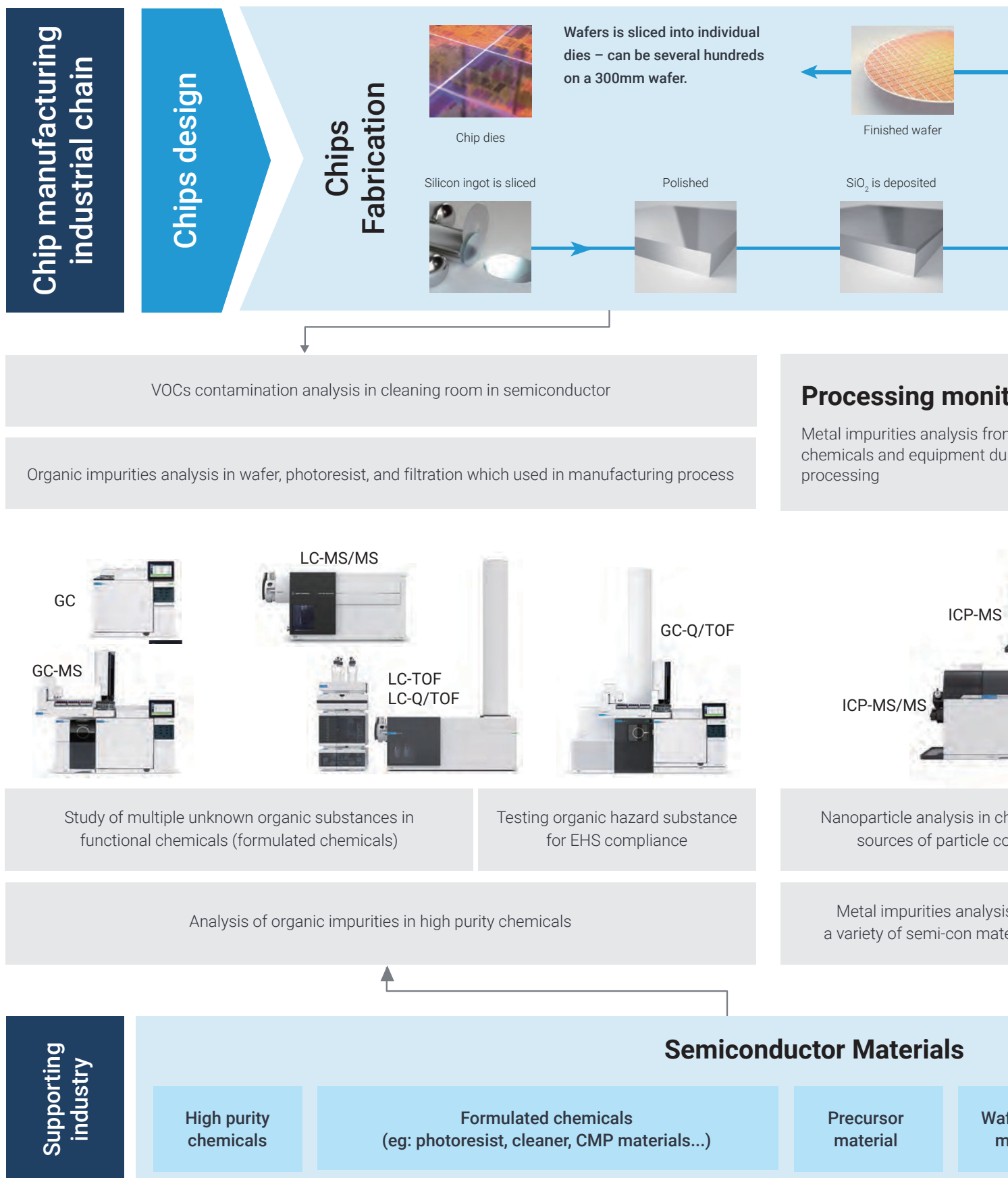
From the 1970s to today, chip manufacturing process has transitioned from the “micrometer” era to the “nanometer” era and node size that less than 10 nm have been developed (now 3nm in the latest chips made by TSMC & Samsung). With the shrinking of components to the nanoscale, control of contaminants and impurities has become more important as even ultra-trace contaminants will decrease yield and cause decrease of product reliability or product malfunction. Impurity analysis of semiconductors and electronic products must cover the various stages of the manufacturing process from testing wafer, raw materials, and process chemicals, to the quality assurance/quality control of the final product. Furthermore, vacuum control is also extremely crucial in device manufacturing, sensor manufacturing, controller manufacturing, and other advanced manufacturing steps in the entire semiconductor industry chain.

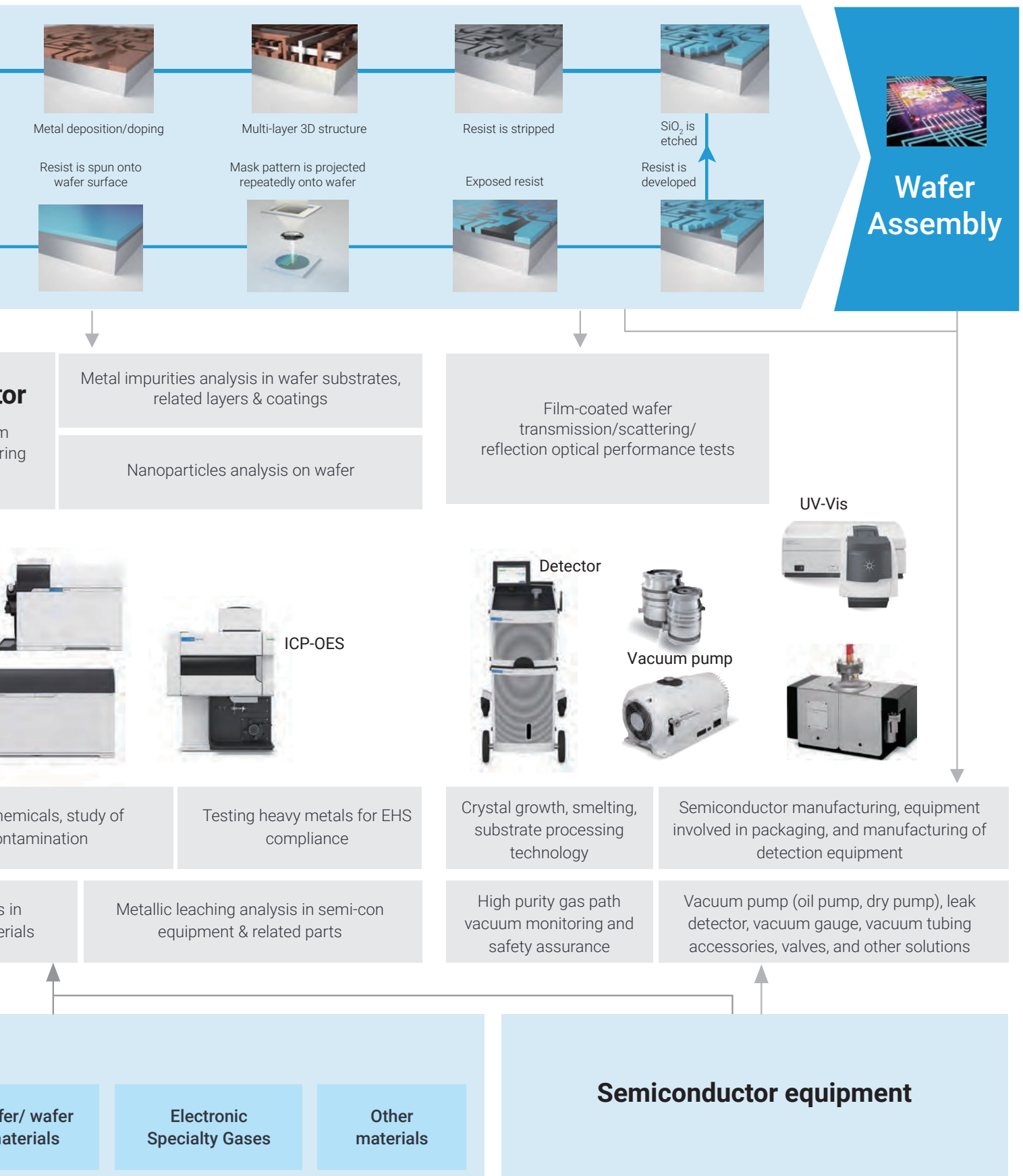
Since the late 1980s, Agilent has been collaborating closely with leading semiconductor manufacturers and chemical suppliers to develop analytical and monitoring technologies for the semiconductor industry chain, address the analytical challenges in the semiconductor industry, and continuously explore the forefront of innovation.

Agilent has accumulated many innovative technologies and outstanding service capabilities. Agilent can provide outstanding analysis device, software, services and support in various aspects of the semiconductor industry chain, such as process monitoring, quality control of raw materials, detection of inorganic impurities, nanoparticles and organic impurities, compliance with environmental health and safety regulations, and vacuum leak detection to help you achieve success.



# Agilent Semiconductor Industry Solutions

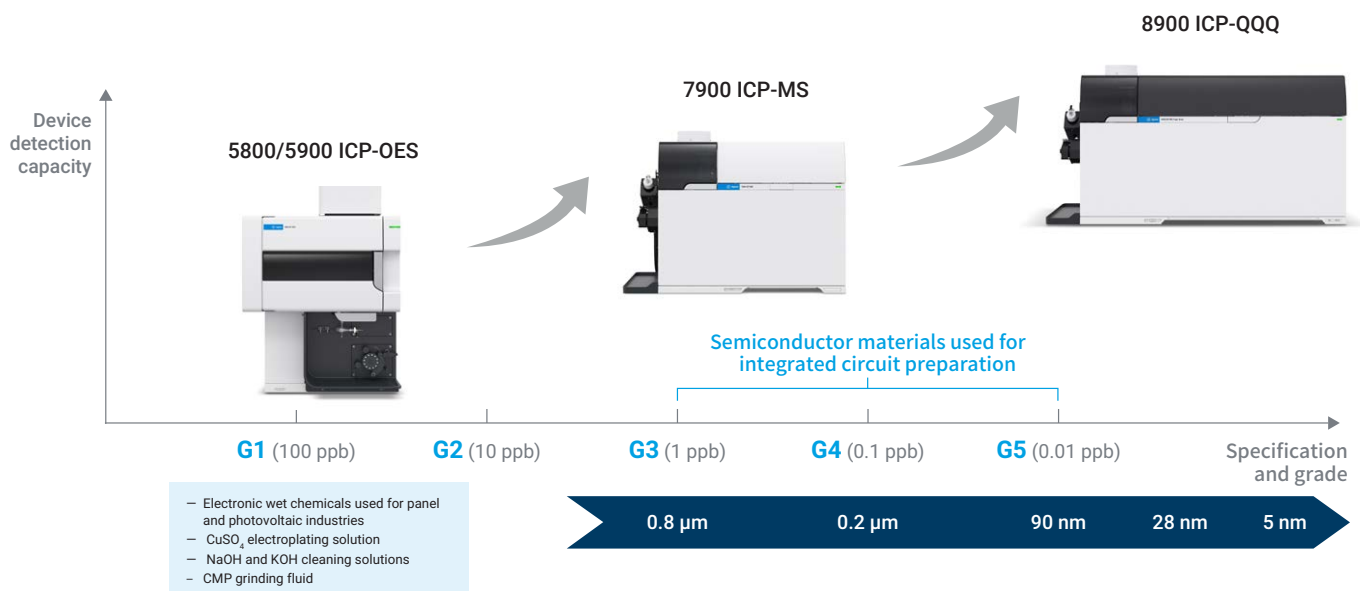




# Typical element analysis scenarios in semiconductor manufacturing and Agilent's solutions



- Monitoring of trace contaminants in chemical used in silicon wafer cleaning and etching
- Monitoring of contaminants in chemical used in wafer/IC manufacturing
- Evaluation of metal contamination in silicon wafer substrate, related layers and coating layers used in silicon
- Analysis of metallic nanoparticle (NP) in chemicals and in wafer processing and cleaning bath
- Metallic contaminants in electrochemical/special gas in the semiconductor supply chain (GC/ICP-MS, GED/ICP-MS)
- Analysis of utilities (eg: incoming water), waste streams, etc.



# Our continuous innovation is driven by 30 years of experience in semiconductor element analysis

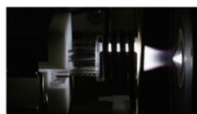
ICP-MS has been used as a metallic element analyzer in the semiconductor industry chain since the 1980s. With continuous iterations in the semiconductor manufacturing process, the entire industry has proposed higher requirements for ICP-MS performance. Since last three decades, Agilent's ICP-MS global R&D center in Tokyo, Japan, has worked closely with customers in the semiconductor industrial chain and led the continuous innovation of ICP-MS in the global semiconductor industry chain

**30**<sup>+</sup>  
years

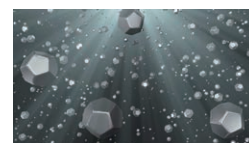
## Innovation milestones of Agilent ICP-MS in the semiconductor industry



Agilent introduced cool plasma technology for the **semiconductor industry** and 4500 ICP-MS, the smallest ICP-MS, manufactured by Agilent started to be used in the clean rooms of various semiconductor factories.



A fully stainless steel base was used and **7700s ICP-MS assembled in the clean room** was used for the semiconductor industry to achieve ppt detection capacity.



Agilent applied single nanoparticle multi-element analysis in chemical quality control in the semiconductor industry.

1992

2003

2009

2012 and 2016

2020

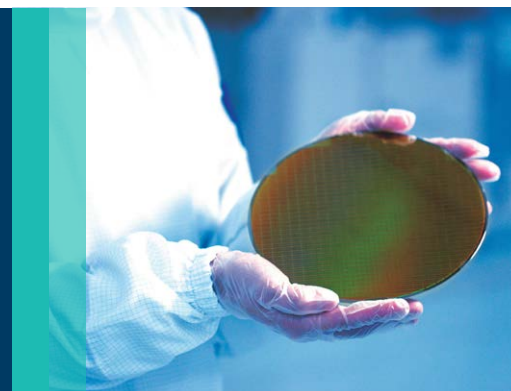
Agilent launched 7500cs ICP-MS specifically for **semiconductors** and combined collision/reaction cell technology to help semiconductor manufacturing enter the nm stage from the  $\mu\text{m}$  stage.



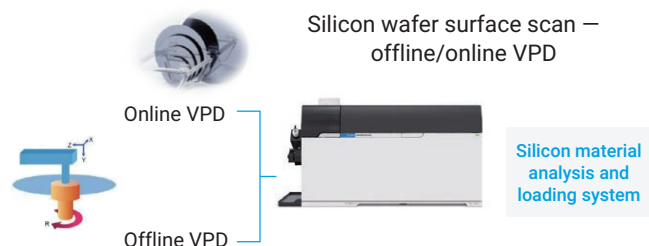
Agilent launched **first-generation and second-generation ICP-MS/MS** with strong de-interference capacity, which were widely used by leading semiconductor companies worldwide for contamination control and increasing yield in advanced manufacturing processes.



# Typical cases of semiconductor industry element analysis solutions



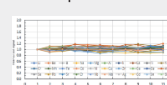
## Silicon wafers



### Silicon wafer surface metal concentration measurement

12 inches	✓	1.0E+07–1.0E+08 atoms/cm <sup>2</sup>
8 inches	✓	1.0E+08–1.0E+09 atoms/cm <sup>2</sup>

Silicon wafer test high stability One standard curve for whole-day stable operation



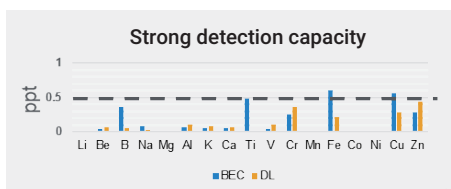
## Ultra-pure chemicals



Inorganic acids: HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, HF, H<sub>2</sub>O<sub>2</sub>

Bases: NH<sub>4</sub>OH, TMAH

Organic reagents: OK73(EBR), IPA



### G5 and higher grade

#### Element content

- Acid or base: < 1 ppt
- Sulfuric acid and organic compounds: < 10 ppt



#### Nanoparticles

- Involved elements: Al, Cr, Fe, Zn, Si, etc.
- Target particle size: < 15 nm

## Semiconductor grade photoresists and accessory reagents

### Photo-resist



Electronic grade NMP or PGMEA dissolved sample

ArF/EUV

ppt level

g line/i line/KrF

ppb level



ICP-MS/MS



ICP-MS

## Electroplating/basic wash solution



ICP-OES

### Dilution at low dilution factor followed by direct analysis

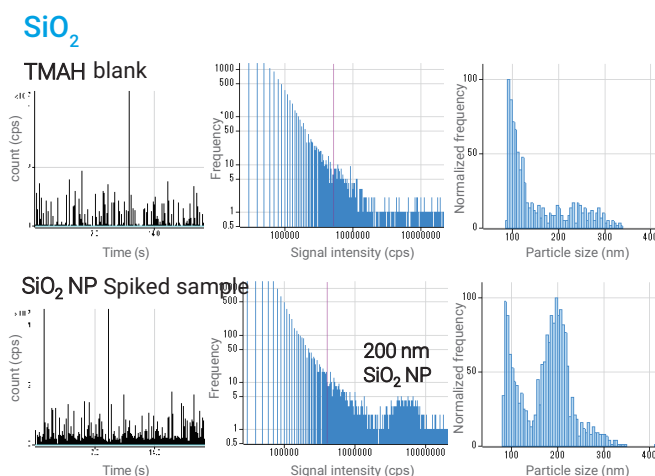
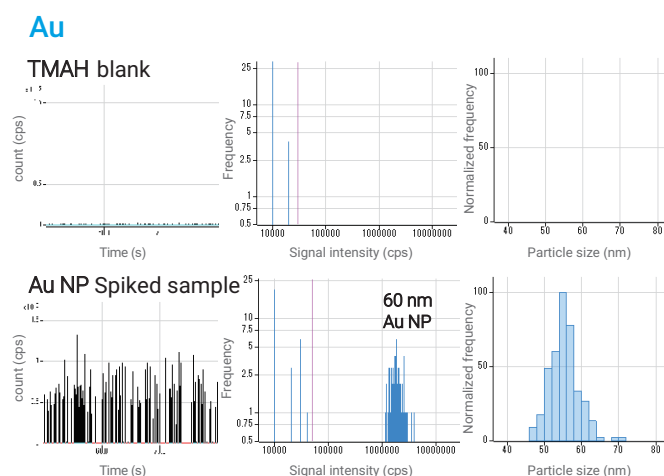
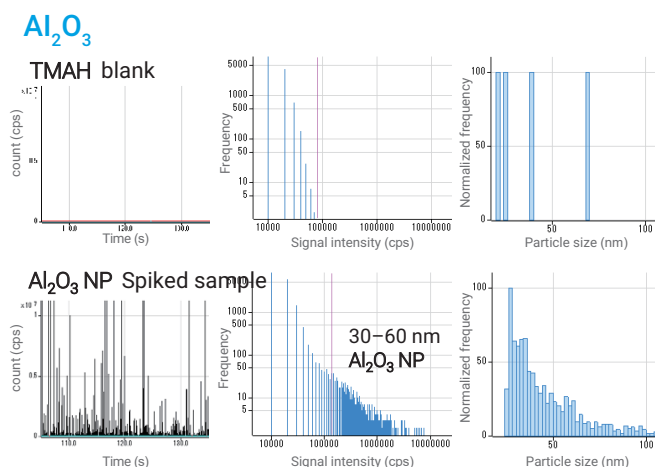
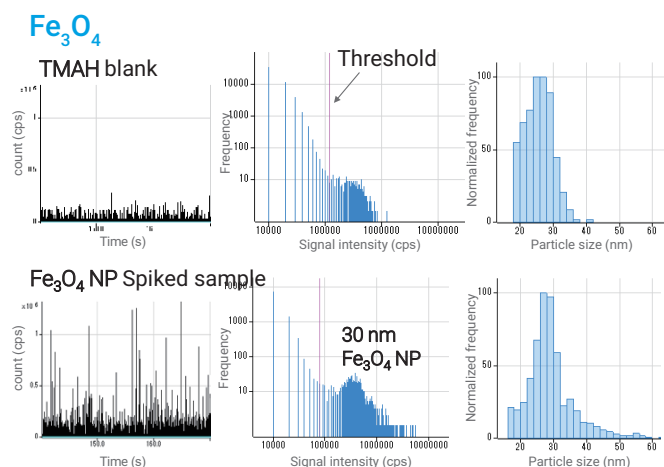
Solution label	Ag 328.068 nm ppb	Al 396.152 nm ppb	As 188.980 nm ppb	Ba 455.403 nm ppb	Be 313.167 nm ppb	Ca 317.933 nm ppb	Cd 228.402 nm ppb	Cu 230.786 nm ppb
489KOH	<5	5.7	<20	27.7	1.1	33.2	2.4	4.8
489KOH	<5	7.3	<20	29.9	1.2	35.6	1.8	4.4
489KOH	<5	6.6	<20	31.8	1.2	36.1	1.8	3.0
489KOH	<5	6.6	<20	31.8	1.2	38.1	2.1	4.6
489KOH	<5	4.8	<20	32.6	0.9	40.0	1.5	2.8
489KOH	<5	6.7	<20	33.2	1.2	42.3	2.4	5.5

Agilent's ICP-MS R&D team in Japan has continuously innovated in the last 30 years and led the transition of inorganic element analysis from the ppt grade to the sub-ppq grade in the semiconductor field. With several migrations in the semiconductor industry chain, Agilent has set up the WW teams dedicated to the applications and after-sales support for global semiconductor manufacturing (Taiwan, S.Korea, Japan, mainland of China, US, EMEA, SEA, etc).



## Using single particle ICP-MS (spICP-MS) to measure nanoparticles in semiconductor chemicals

- Multi-element analysis of nanoparticle (NP) is used to study the causes of particle contamination
- Monitoring of metallic NP in chemicals, wafer substrate and device surfaces, and cleaning bath
- MS/MS can achieve low background, high sensitivity, and interference control in multi-element analysis of NP in semiconductor grade chemicals
- Agilent's rapid multi-element nanoparticle analysis software can be used to continuously acquire data of up to 16 types of NPs in a single sample analysis



- Agilent 8900 ICP-MS/MS can be used to measure multi-element NPs in 1% TMAH in the multi-element spICP-MS mode;
- The temporal resolution signal, signal distribution, and particle size distribution map of Fe<sub>3</sub>O<sub>4</sub>, Al<sub>2</sub>O<sub>3</sub>, Au, and SiO<sub>2</sub> NPs are shown above;
- The Fe<sub>3</sub>O<sub>4</sub> and SiO<sub>2</sub> NP results show that the blank TMAH solution analyzed in this study contained these two NPs;
- Small particles (such as 30-nm Fe<sub>3</sub>O<sub>4</sub> NP) could still be detected when large particles (such as 200-nm SiO<sub>2</sub> NP) are present in the solution.

# Automated analysis of metallic contaminants in silicon wafers

Agilent's ICP-MS systems can be integrated to all advanced automated VPD scanners to achieve fully automated analysis of contamination on silicon wafer surfaces

## Vapor phase decomposition

Metallic contamination may be introduced to semiconductor devices during cleaning, etching oxide growth, and ion injection. Trace contaminants may also come from quartzite (quartz sand) used in production of polycrystalline silicon chunks and pure monocrystalline silicon ingots from which the individual wafer blanks are sliced. The main contaminant elements in quartzite are iron, aluminum, calcium, and titanium, and other elements may also be introduced during the carbothermal reduction process of converting quartzite to 98% pure silicon. After the process, vapor phase purification and chemical vapor phase deposition are used to remove most impurities to obtain 9N-11N pure silicon.

Cutting and polishing wafer may also introduce trace elements, such as chemical-mechanical planarization (or polishing) slurry. Most attention is paid to transition metals, alkali and alkaline earth elements, which may not be uniformly distributed in wafers. Iron may spread from the silicon substrate to the surface oxidized layer and titanium impurity levels may change due to segregation during monocrystalline silicon ingot melting and cooling.

In order to ensure that metallic contaminants will not have adverse effects on IC devices, the concentrations of trace metals on wafer surfaces must be monitored and controlled. The bare silicon layer on wafer surfaces is rapidly oxidized to  $\text{SiO}_2$  when exposed to oxygen and water in the atmosphere. this natural oxide layer is less than 2 nm thick (one  $\text{SiO}_2$  molecule). If an insulation film is required in the IC design, the wafer is heated to 900–1200 °C in the presence of  $\text{O}_2$  or steam, thereby forming a thicker oxide layer on the wafer surface. The thickness of this thermal oxide layer is up to 100 nm (0.1  $\mu\text{m}$ ). Vapor phase decomposition (VPD) combined with ICP-MS can be used to measure extremely low concentrations of trace metals in natural and thermal oxidized  $\text{SiO}_2$ .

Agilent ICP-MS and ICP-MS/MS devices are compatible with all mainstream VPD systems, including:

- Elemental Scientific Inc. (US)
- IAS Inc. (Japan)
- PVA TePla AG (Germany)
- NvisANA Co. Ltd (South Korea)
- NAS GIKEN (Japan)



WCS M300 automated VPD scanner system from NvisANA (South Korea)

## Combination of ICP-MS and vapor phase decomposition

VPD-ICP-MS is a validated method for measuring trace metallic contaminants in silicon wafers. VPD wafer sampling has good sensitivity as it can concentrate metals on large surface oxide layers to a single liquid droplet for measurement.

This process (can be fully automated) consists of 4 steps:

1. The silicon wafer is placed in the VPD chamber and exposed to HF vapor to decompose the natural oxide or thermal oxide  $\text{SiO}_2$  surface layer.
2. An extraction droplet (usually 250  $\mu\text{L}$  of 2% HF/2%  $\text{H}_2\text{O}_2$ ) is placed on the wafer and the wafer is tilted in a precisely controlled manner so that the droplet “sweeps” the surface of the wafer.
3. As the extraction droplet moves on the wafer surface, it will collect the residue from the  $\text{SiO}_2$  decomposition, along with any metallic contaminants.
4. The extraction droplet is transferred from the wafer surface to ICP-MS or ICP-MS/MS system for analysis

## Benefits of combining ICP-MS or ICP-MS/MS and VPD

VPD can be manually performed. However, only experienced operators can achieve consistent soluble metal recovery from the  $\text{SiO}_2$  layer. VPD can also be used together with various element analysis techniques for quantitative analysis of metallic contamination. However, ICP-MS or ICP-MS/MS can provide high sensitivity and low limit of detection for all essential analytes. At the same time, the automated VPD process can ensure consistency and decrease the possibility of contamination.

The Agilent 7900 and 8900 ICP-MS analyzers can be integrated with VPD systems for fully automated analysis of metallic impurities in silicon wafers. Both of the two Agilent ICP-MS provide the good matrix tolerance required to analyze thermally oxidized  $\text{SiO}_2$ , of which  $\text{SiO}_2$  matrix concentration in the extraction droplet can reach 5000 ppm (depending on the thickness of oxide layer). Agilent 8900 also has the advantage of much higher sensitivity, lower background, and MS/MS operation for the most effective interference removal compared with other ICP-MS systems. These capabilities enable the 8900 to achieve lower limits of detection and improved accuracy.

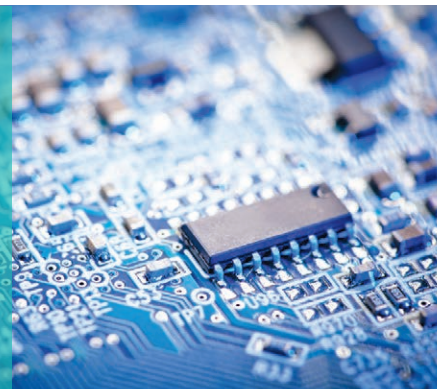


Munich Metrology fully automated wafer surface measurement system (WSMS) manufactured by PVA TePla that was integrated with Agilent 8800 ICP-MS/MS



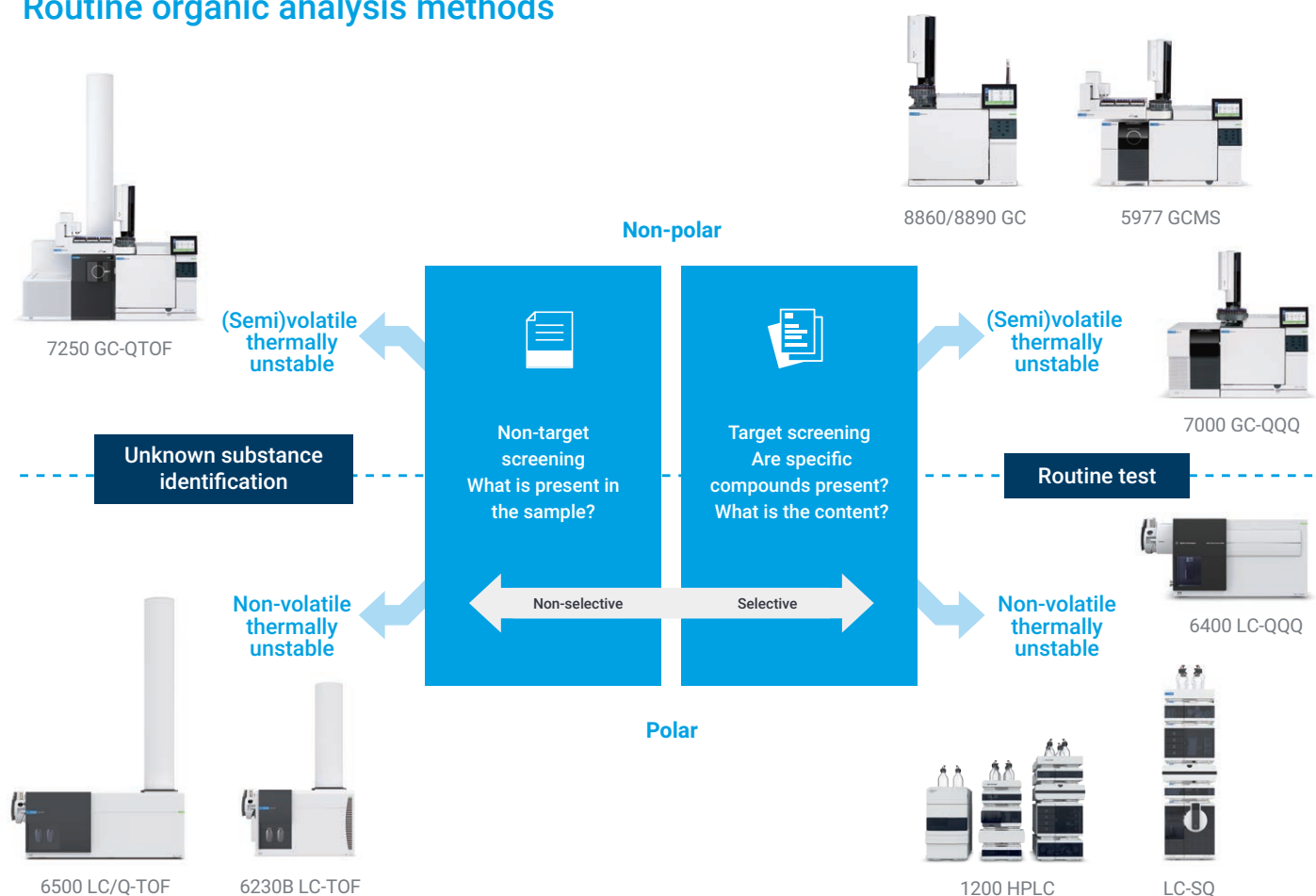
Expert PS fully automated VPD-ICP-MS system manufactured by IAS Inc. that was integrated with Agilent 8800 ICP-MS/MS. The figure was provided by ST Microelectronics (Crolles, France)

# Semiconductor industry organic analysis solutions



- Organic contaminants analysis on wafer surfaces
- Organic impurities analysis in high purity chemicals and UPW
- Impurity analysis in cleaning/ stripping/ plating solution
- Organic composition analysis in chemicals, formulation analysis (eg: formulation of photoresist, CMP slurry, etc)
- Contaminant control of filters and other semiconductor process parts
- Analyses of volatile organic contaminants in the semiconductor clean room
- Measurement of harmful organic substances in electronic gases according to regulatory requirements (e.g. RoHS, REACH)

## Routine organic analysis methods





Innovation never stops — Agilent: leader in gas chromatography.



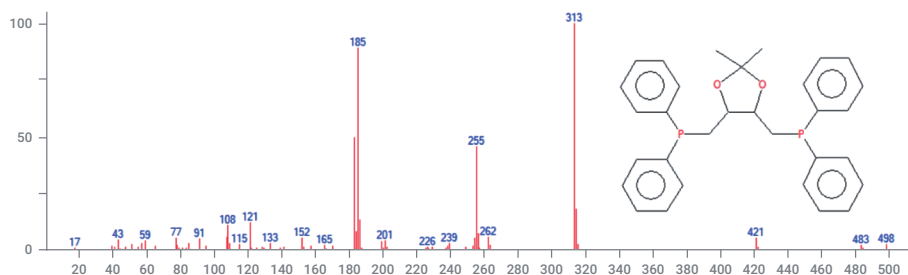
### Latest Intelligent Connected GC System



Solutions with stable and reliable quality

## Organic analysis solutions for semiconductor industry – typical case 1

- High resolution, high sensitivity chromatography and mass spectrometry to assist in identification of unknown impurities
- Quantitative control of impurities in propylene glycol methyl ether (PGME)/propylene glycol methyl ether acetate (PGMEA)



Gas chromatography-high resolution mass spectrometry (GC-QTOF) is used to identify trace impurities



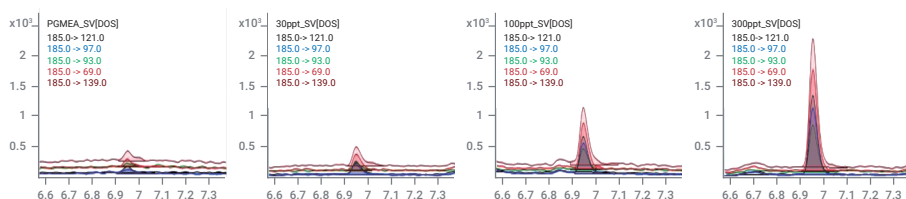
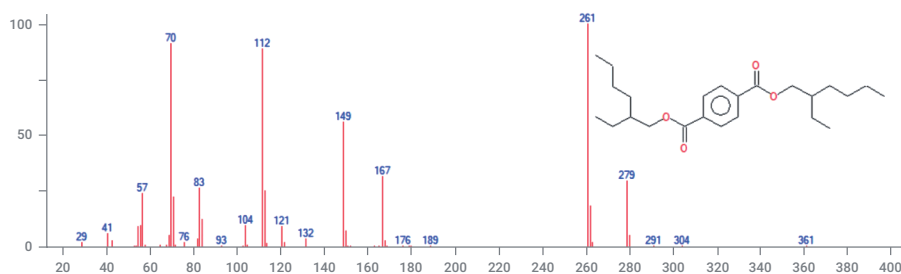
Some of the impurities identified were phthalate plasticizers, di-iso-octyl phthalate (DIOP) and bis(2-ethylhexyl) terephthalate (DOTP)



GC/MS(MS) is used for accurate quantitation of identified trace impurities



ppt grade or even ppt level plasticizer impurities could be detected without concentration



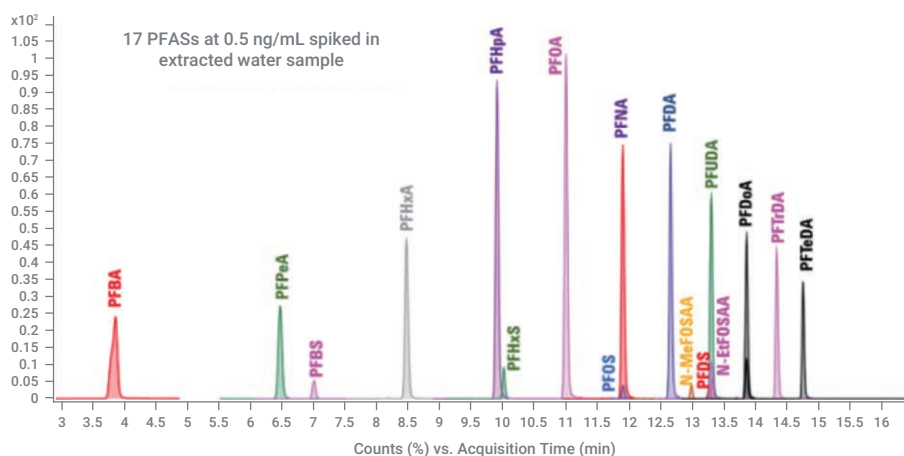
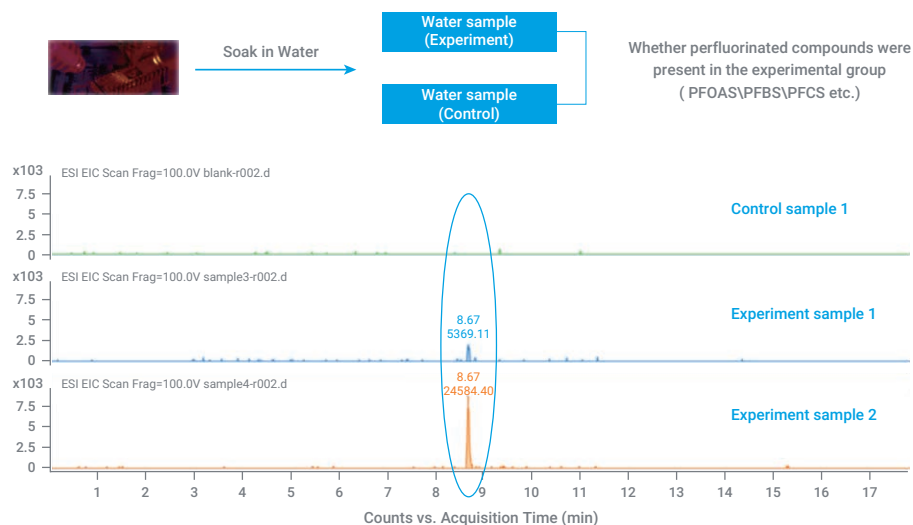
7250GC/Q-TOF



7000GC-QQQ

## Organic analysis solutions for semiconductor industry – typical case 2

- Study of migrating substances in water from submerged ArF photoresists
- The main substance causing harmful migration in photoresists is photo acid generator (PAG) and its main anion is PFAS compound
- Excessive amount of migrating substances will decrease yield (e.g. Watermark effect and T-topping effect), and cause damage to the lithography machine lens
- Using LC-QQQ for qualitative and quantitative analysis of PPAS acid generators



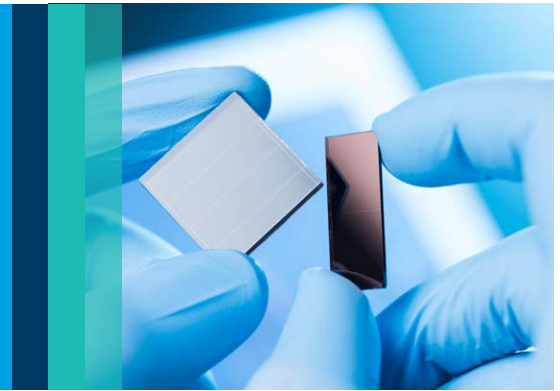
### Instruments



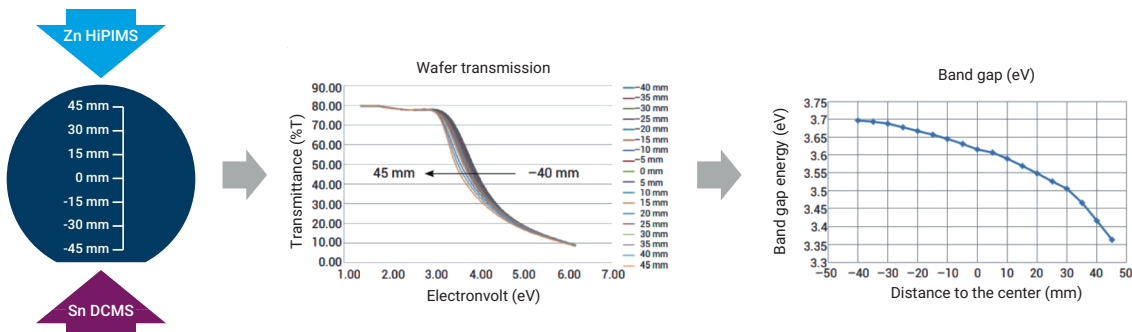
### Consumables



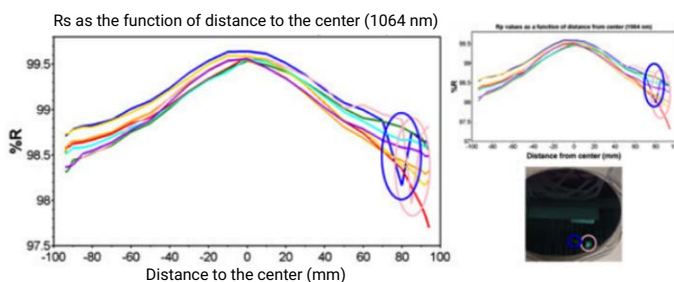
# Optical performance analysis of semiconductor materials



- Film-coated wafer transmission/scattering/reflection optical performance test of terminal equipment such as CPU, RAM
- Wafer coating layer surface optical performance characterization and evaluation of coating layer uniformity
- The optical performance test results are used to determine and overcome potential variability in the coating layer process



- Agilent Cary 7000 universal measurement spectrophotometer (UMS) with solid autosampler is used to analyze the optical performance of coating layer wafers
  - Deposition direction schematic diagram and coordinate system of wafer direction. DCMS deposition is used for Sn and HiPIMS deposition is used for Zn. A 5 mm interval is used for spectrophotometry at the -40 mm to 45 mm interval to obtain the transmission spectrum of 11 sites on the entire wafer diameter.
  - Through acquisition of the transmission spectrum, the band gap energy of the ZTO substrate is mapped to the entire diameter range of the wafer.
  - The data shows some differences, such as the deposition process causing Zn concentration on the wafer apex to be the highest, therefore frequency was the lowest.



- Cary 7000 UMS was used for reflectivity test of the optical wafer at 1064 nm
- The distance from the measurement point to the center of the optical wafer was plotted. The results showed that:
  - Reflectivity decreases from the center to the edge of the wafer. The high similarity and consistency of the different curves showed that the wafer has centrally symmetrical optical contours
  - Subsequent visual inspection shows that abnormal Rs and Rp values at the 80 mm diameter of the 90° string and 85 mm of the 67.5° string were due to wafer surface contamination



# Vacuum solutions



Serves the entire industrial chain of the semiconductor market and upstream suppliers. Provides vacuum pump (oil pump, dry pump), leak detector, vacuum gauge, vacuum tubing accessories, and valves. The scope of application includes:

- Semiconductor manufacturing, packaging equipment, and detection equipment
- Crystal growth and extension equipment
- High purity gas tube test and safety assurance
- Sensor air tightness test

## Vacuum Pump

Agilent provides vacuum systems for mass spectrometers of many well-known brands. As the leader in oil-free detection equipment, Agilent has used oil-free vacuum pumps in GC/MS, GC/QQQ, and LC/MS systems. At the same time, we also provide many vacuum generation and measurement devices for rough vacuum to high vacuum and ultra-high vacuum for laboratories and many industry applications.

## Vacuum leak detection

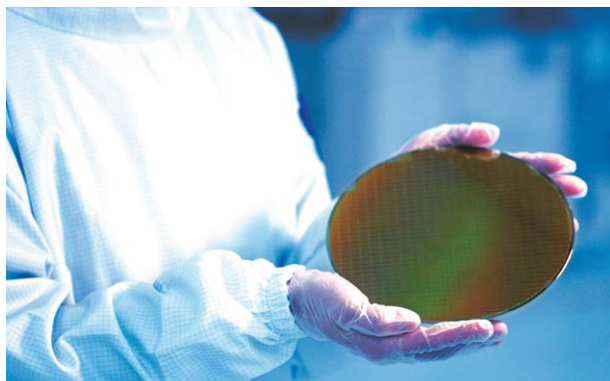
The HLD leak detector perfectly combines the ease of use of color touchscreen operation and intelligent advanced expert system. The dry leak detector equipped with a dry backing pump is easy to transport and maintain, and is an essential tool for high frequency helium gas leak detection.

## Vacuum measurement

Agilent provides many active and passive vacuum gauges and controllers for accurate and reliable vacuum control and measurement at atmospheric pressure to UHV/XHV. This includes thermocouple (TC), B-A (TA), Pirani, inverted magnetron (IMG), and hot filament iron gauge (HFIG) vacuum gauges. Agilent vacuum controllers are compatible with Agilent vacuum gauges and vacuum gauges from similar manufacturers, and can process up to 12 channels. Agilent controllers have multiple communication ports, including Profibus, Ethernet, and RS-232/485 (other ports can be requested on demand).



## Inorganic Impurities in Semiconductors Applications



### Wafer & Semiconductor Materials Impurities Analysis

Monitoring and controlling trace-level contamination in wafer, CMP slurries, interconnects, REE materials, and photoresist chemicals is critical.

[Learn more](#)



### Semiconductor Process Chemicals Impurities Analysis

Trace-level contaminants have detrimental effects on manufacturing. Learn more about testing for analysis of impurities in semiconductor process chemicals.

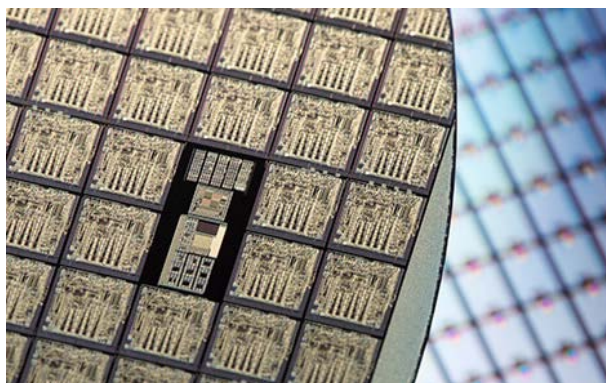
[Learn more](#)



### Electronic Gases Impurities Analysis

Novel, efficient, and elegant approaches for direct analysis of semiconductor impurities testing in electronic gases.

[Learn more](#)



### Particle Analysis in Semiconductors

Solutions for monitoring metallic nanoparticles (NPs) and dissolved metals in bulk chemicals and in wafer processing and cleaning baths.

[Learn more](#)

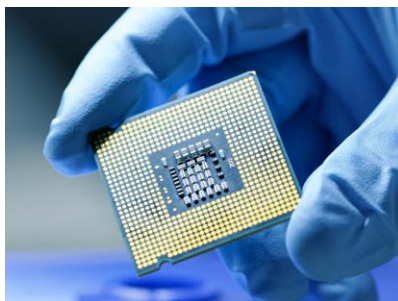
## Semiconductors & Electronics Testing Applications



### Inorganic Impurities in Semiconductors

Monitor and control trace and ultra-trace-level metal contaminants in wafers, sputtering targets, process chemicals, photoresists, and electronic gases.

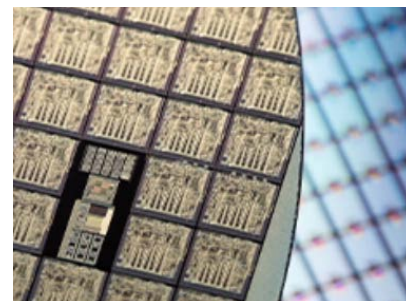
[Learn more](#)



### Organic Impurities in Semiconductors

Performing analysis of trace-level organic impurities? With industry-leading instrumentation and applications, Agilent has you covered.

[Learn more](#)



### Particle Analysis in Semiconductors

Solutions for monitoring metallic nanoparticles (NPs) and dissolved metals in bulk chemicals and in wafer processing and cleaning baths.

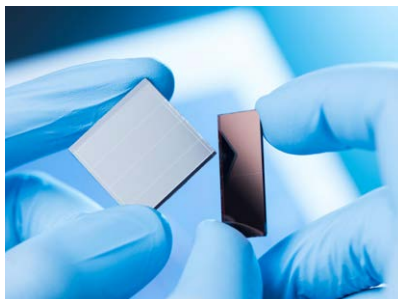
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### Environmental Health & Safety Compliance for Materials

Agilent provides Environmental Health and Safety (EH&S) testing technologies to support industry, helping to ensure compliance with EH&S regulations.

[Learn more](#)



### Photonics & Optoelectronics Devices & Components Analysis

Agilent UV-Vis and UV-Vis-NIR spectrophotometers provide complete photonics test and measurement solutions for photonic devices and components.

[Learn more](#)



### Vacuum & Leak Detection in Semiconductors & Electronics

Vacuum pumps and gauges and precise, robust, easy-to-use leak detectors save you time and money.

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