Semiconductor production

Analytical methods for critical QC parameters

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The production of semiconductors requires high quality standards, as contaminants can cause defective wafers. There are several steps involved in processing the raw material (monocrystalline silicon) into a finished integrated circuit. Each step requires a significant amount of attention to detail to ensure that the quality of the finished product is high.

Monitoring the water purity, acids in etching baths, metals content in plating baths, or ionic impurities is crucial to optimize the semiconductor production. Metrohm provides top-quality analytical instruments, know-how, and first-class, on-site service to enable and support your production of semiconductors.

The table below serves as an overview, of several parameters of interest that can be analyzed by different techniques using Metrohm instrumentation. More detailed information is found in the corresponding linked Metrohm application documents. Missing your application here? Please contact your local Metrohm organization to discuss possible solutions.

Process step	Parameter and Matrix	Standard	Analysis technique	Application document
Wafer testing	Wafer and film analysis		Raman spectroscopy	
CMP polishing	H_2O_2 in CMP slurries		Titration, NIR spectroscopy, Process analysis	AN-PAN-1054
	Conductivity of CMP slurries	SEMI C99	Conductivity	AB-102
	pH value of CMP slurries	SEMI C101	pH measurement, Process analysis	AB-188
SC1/SC2 (Standard cleaning 1 and 2)	NH_4OH and H_2O_2 in SC1 (APM) baths		Titration, NIR spectroscopy, Raman spectroscopy, Process analysis	AN-PAN-1055
	HCl, H_2O_2 in SC2 baths		Titration, NIR spectroscopy, Process analysis	AN-PAN-1055
	Trace transition metals (Fe, Ni, Cu) in SC2 (D-clean) baths		Ion chromatography	
Photolithography	Acidic and basic gases as airborne molecular contaminants (AMC)		Ion chromatography, Process analysis	
	Total alkalinity, total normality in alkaline developer solutions	SEMI P11	Titration	
	Water in photoresist	SEMI P8	Karl Fischer titration	
	Trace chloride, sulfate in photoresists developers		Ion chromatography	
	TMAH in photoresists developers		Titration, Raman spectroscopy, Process analysis	AN-PAN-1028, AN-T-234
	Ultratrace anions in tetramethylammonium hydroxide (TMAH)		Ion chromatography, Process analysis	AN-S-250
Etching	Anionic traces (F^-, acetate, formate, Cl^-, NO_2^-, Br^-, NO_3^-, PO_4^{3-}, SO_4^{2-}) in ammonium hydroxide	SEMI C21	Ion chromatography	AN-S-365, AN-S-393
	Assay of ammonium hydroxide	SEMI C21	Titration	
	Nitric acid, hydrofluoric acid, hexafluorosilic acid in etching baths		Titration, Process analysis	AN-H-139, AB-344
	Hydrofluoric acid, nitric acid in etching baths (PETCH baths)	SEMI C34	Titration, NIR spectroscopy, Process analysis	AN-H-138
	Acetic acid, phosphoric acid, nitric acid in phosphoric etchants	SEMI C37	Titration, Process analysis	AN-H-016, AN-T-234
	Nitric acid, phosphoric acid, sulfuric acid, hydrofluoric acid in etching solutions		Titration, NIR spectroscopy, Raman spectroscopy, Process analysis	AN-NIR-090, WP-067
	Sulfuric acid and hydrogen peroxide (H_2O_2) in photoresist stripping (SPM baths)		Titration, NIR spectroscopy, Raman spectroscopy, Process analysis	AN-PAN-1062
	Sulfuric acid and hydrogen peroxide (H_2O_2) in DSP and DSP+ baths		Raman spectroscopy, Process analysis	AN-PAN-1062

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Process step	Parameter and Matrix	Standard	Analysis technique	Application document
Etching (continued)	Acetic acid, hydrofluoric acid, nitric acid in etching solutions	SEMI C34	Titration, NIR spectroscopy, Raman spectroscopy, Process analysis	AN-NIR-091, WP-067
	Anions (F^-, acetate, NO_3^-, SO_4^{2-}, silicate, hexafluorosilicate, PO_4^{3-}, monofluorophosphate) acid in etching solutions		Ion chromatography	
	Zn, Cd, Pb, Ni, and Co in hydrochloric acid		Voltammetry	AN-V-028
	Trace nitrate in phosphoric acid	SEMI C36	Ion chromatography	
	Iron (Fe) in phosphoric acid		Voltammetry	AN-V-129
	Zn, Cd, Pb, Ni, and Co in sodium hydroxide		Voltammetry	AN-V-010
	Fe, Ni, and Co in sulfuric acid		Voltammetry	AN-V-131, AN-V-132
	pH value of etching solution		pH measurement	AB-188
Damascene Metallization / Plating	Chloride in acid copper baths		Titration	AN-T-100
	Sulfuric acid and copper in acid copper baths		Titration, Process analysis	AN-T-223
	Cyanide in alkaline copper baths		Titration, Raman spectroscopy (SERS)	AB-046
	Fundamental studies of electrodeposition of copper onto PCB plates using electrochemical quartz cristal microbalance (EQCM)		Electrochemistry	AN-EC-011
	Electrochemical impedance spectroscopy (EIS) as fundamental research tool for the characterization of coatings, metal deposition, and conductivity		Electrochemistry	AN-EIS-001, AN-EIS-002, AN-EIS-003, AN-EIS-004, AN-EIS-005, AN-EIS-009
	Suppressors, brighteners, and levelers in acid copper baths		CVS, Process analysis	WP-051, AN-PAN-1067
Die Attach	Anions and cations in die attach adhesives		Ion chromatography	
Leadframes	Ionic contamination in leadframes and leadframe interleafing	SEMI G52, SEMI G64	Ion chromatography	
Wire Bonding	Nickel and hypophosphite in electroless nickel plating baths		Titration, Process analysis	AN-PAN-1012

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Process step	Parameter and Matrix	Standard	Analysis technique	Application document
Advanced Wafer Level Packaging (UBM, Solder Bump, Copper Pillar, RDL, TSV) bumping	Suppressors, brighteners, and levelers in acid copper baths, tin baths, tin/lead baths, tin/silver baths		CVS, Process analysis	WP-051, AN-PAN-1067
	pH value in nickel plating baths		pH measurement, Process analysis	AB-188
	Nickel and boric acid in nickel plating baths		Titration, Process analysis	AB-195
	Copper, sulfuric acid, and chloride in acid copper baths		Titration, Process analysis	AN-T-100, AN-T-223
	Tin and silver in tin/silver baths		Titration, Process analysis	
	Nickel and hypophosphite during electroless nickel plating in immersion deposition process across under bump metallization (UBM)		Titration, Process analysis	AN-PAN-1012
	Palladium during electroless palladium plating in immersion deposition process across under bump metallization (UBM)		Process analysis	
	Hypophosphite during electroless palladium plating in immersion deposition process across under bump metallization (UBM)		Titration, Process analysis	
	Gold and nickel during gold immersion in immersion deposition process across under bump metallization (UBM)		Titration, Process analysis	
	Gold and thallium in non-cyanide based gold bump electroplating process		Voltammetry, Process analysis	AN-V-199
	Sulfite in non-cyanide based gold bump electroplating process		Titration, Process analysis	
Encapsulation	Quality control of laminates		NIR spectroscopy	AN-NIR-089
	Absence of halogens in PCB basic materials	IEC &1249-2-21	Ion chromatography	AN-CIC-015
	Ions on the surface of PCBs		Ion chromatography	AN-S-317, AN-C-149
Blending and QC of etchants	Anionic traces (F^-, acetate, formate, Cl^-, NO_2^-, Br^-, NO_3^-, PO_4^{3-}, SO_4^{2-}) in ammonium hydroxide	SEMI C21	Ion chromatography	AN-S-365, AN-S-393
	Assay of ammonium hydroxide	SEMI C21	Titration	
	Hydrofluoric acid, nitric acid in etching baths (PETCH baths)	SEMI C34	Titration, NIR spectroscopy, Process analysis	AN-H-138
	Acetic acid, phosphoric acid, nitric acid in etching solutions		Titration, NIR spectroscopy, Process analysis	AN-H-016, AN-T-234
	Nitric acid, phosphoric acid, sulfuric acid, hydrofluoric acid in etching solutions		Titration, NIR spectroscopy, Raman spectroscopy	AN-NIR-090, WP-067
	Acetic acid, hydrofluoric acid, nitric acid in etching solutions		Titration, NIR spectroscopy, Raman spectroscopy, Process analysis	AN-NIR-091, WP-067

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Process step	Parameter and Matrix	Standard	Analysis technique	Application document
Blending and QC of etchants (continued)	Anions (F^-, acetate, NO_3^-, SO_4^{2-}, silicate, hexafluorosilicate, PO_4^{3-}, monofluorophosphate) acid in etching solutions		Ion chromatography	
	Zn, Cd, Pb, Ni, and Co in hydrochloric acid		Voltammetry	AN-V-028
	Iron (Fe) in phosphoric acid		Voltammetry	AN-V-129
	Total acidity, phosphoric acid in phosphoric etchants	SEMI C37	Titration	
	Zn, Cd, Pb, Ni, and Co in sodium hydroxide		Voltammetry	AN-V-010
	Fe, Ni, and Co in sulfuric acid		Voltammetry	AN-V-131, AN-V-132
Purity of raw materials (solvents, etc.)	Assay of ammonium fluoride (40%)	SEMI C20	Titration	
	Anions and cations in ammonium fluoride (40%)	SEMI C20	Ion chromatography	
	Ultratrace anions in cyclopentanone (CPN)		Ion chromatography	
	Cd, Co, Cu, Fe, Ni, Pb, and Zn in electronic grade materials		Voltammetry	AB-147
	Ultratrace anions in hexamethyldisilazane (HMDS)		Ion chromatography	
	Assay of hydrochloric acid	SEMI C27	Titration	
	Trace anions in hydrochloric acid		Ion chromatography	
	Assay of hydrofluoric acid	SEMI C28, SEMI C29	Titration	
	Trace anions in hydrofluoric acid	SEMI C28, SEMI C29	Ion chromatography	
	Assay of hydrogen peroxide	SEMI C30	Titration	
	Trace trimethylamine (TMA) and trace cations (Li^+, Na^+, NH_4^+, K^+, Mg^{2+}, Ca^{2+}) in hydrogen peroxide		Ion chromatography	AN-CS-012, AN-CS-019
	Trimethylamine (TMA) and NH_4^+ in hydrogen peroxide		Raman spectroscopy (SERS)	
	Organic acids (formate, acetate, propionate, succinate, malonate, oxalate, butyrate) in hydrogen peroxide		Ion chromatography	WP-086
	Trace anions (F^-, Cl^-, NO_2^-, Br^-, NO_3^-, PO_4^{3-}, SO_4^{2-}) and pyrophosphate in hydrogen peroxide	SEMI C30	Ion chromatography	AN-S-393 (only trace anions), AN-S-352 (pyrophosphate + anions)

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Process step	Parameter and Matrix	Standard	Analysis technique	Application document
Purity of raw materials (solvents, etc.) (continued)	Water content in solvents (e.g., IPA)		Karl Fischer titration, NIR spectroscopy, Process analysis	AB-137, AB-077
	Ultratrace anions in isopropanol (2-propanol, IPA)	SEMI C41	Ion chromatography	WP-086
	Acidity, alkalinity, pH value in liquid chemicals	SEMI C1	Titration, pH measurement	
	Ultratrace anions in methanol		Ion chromatography	
	Assay of nitric acid	SEMI C35	Titration	
	Trace anions (F^-, Cl^-, PO_4^{3-}, SO_4^{2-}) in nitric acid		Ion chromatography	AN-S-344, 8.000.6073
	Ultratrace anions in *N*-methyl 2-pyrrolidone (NMP)	SEMI C33	Ion chromatography	WP-086
	Trace nitrate in phosphoric acid	SEMI C36	Ion chromatography	
	Ultratrace anions in propylene glycol methyl ether (PGME)		Ion chromatography	
	Trace anions in propylene glycol methyl ether acetate (PGMEA)		Ion chromatography	
	Assay of sodium hydroxide	SEMI C43	Titration	
	Assay of sulfuric acid	SEMI C44	Titration	
	Trace ammonium in sulfuric acid		Ion chromatography	
	Assay of tetramethylammonium hydroxide (TMAH)		Titration	
	Ultratrace anions in tetramethylammonium hydroxide (TMAH)		Ion chromatography	AN-S-250
Purity of material used during processing	Ionic contamination of polymer materials and components used in ultrapure water and liquid chemical distribution systems	SEMI F57, ASTM D4327	Ion chromatography	
Purity of water	Ultratrace cations (Li^+, Na^+, NH_4^+, K^+, DEA, Zn^{2+}, Mg^{2+}, Ca^{2+}) in high purity water	SEMI F63, SEMI F75	Ion chromatography, Process analysis	AN-C-159, AN-CS-013
	Ultratrace anions (F^-, Cl^-, NO_2^-, Br^-, NO_3^-, SO_4^{2-}, PO_4^{3-}) in high purity water	ASTM D5996, ASTM D5542, ASTM D4327, ASTM D7980, SEMI F63, SEMI F75	Ion chromatography, Process analysis	
	Organic acids (formate, acetate, oxalate) besides standard anions in high purity water	ASTM D5542	Ion chromatography	
	Urea in high purity water		Ion chromatography	
	Silicate, borate in high purity water	SEMI F63	Ion chromatography, Process analysis	AN-N-054, AN-N-005
	Transition metals in high purity water		Ion chromatography, Voltammetry	
	Trimethylammonium (TMA-3), tetramethylammonium (TMA-4) besides cations in high purity water		Ion chromatography	

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Process step	Parameter and Matrix	Standard	Analysis technique	Application document
Chemical recovery and wastewater treatment (CUB, PUB)	Anions, cations, TMAH, and Cr(VI) in wastewater		Ion chromatography	AN-CS-018, AN-S-106, AN-S-107, AN-S-357
	Chemical oxygen demand (COD) in wastewater		Titration, Process analysis	AB-178
	Cd, Co, Cu, Fe, Ni, Pb, Tl, and Zn in wastewater		Voltammetry	AB-231
	Fluoride in hydrofluoric liquid industrial waste system		Ion chromatography, Titration, Process analysis	AB-082, AN-S-186
	Phenol in wastewater		Process analysis	
	Cyanide in wastewater		Ion chromatography, Titration, Ion measurement, Raman spectroscopy (SERS)	AN-P-083, AB-046, AN-I-009
	Ammonia in liquid industrial waste system		Ion measurement, Process analysis	
	Copper in electrowinning process		Process analysis, X-ray fluorescence (XRF)	
	Copper in liquid industrial waste system		Process analysis	
	H_2O_2 and chloride in sulfuric acid recovery systems		Titration, Process analysis	

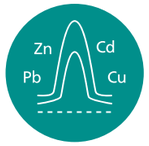
Analysis techniques

TITRATION – SPECIFIC, ACCURATE, AND RELIABLE

Potentiometric titration is well suited for the assay of pure acids and bases and for the determination of the individual concentrations of acids in etchants. It is an inexpensive method which can be automated to optimize laboratory efficiency.

ION CHROMATOGRAPHY – HIGHLY EFFICIENT TRACE ANALYSIS

Ion chromatography (IC) is an efficient and precise multi-parameter method used to quantify ionic impurities in acids, organic solvents, and even high purity water. Using the Metrohm intelligent Preconcentration Technique (MiPCT) enables trace analysis in the ng/L range. MiPCT combined with Inline Matrix Elimination (MiPCT-ME) makes the analysis of challenging matrices possible.



CVS AND VOLTAMMETRY – HIGHLY SENSITIVE ANALYSIS OF ADDITIVES AND TRANSITION METALS

Cyclic Voltammetric Stripping (CVS) and the related Cyclic Pulse Voltammetric Stripping (CPVS) are the standards for analyzing organic additives (i.e., suppressor, brightener, and leveler) in copper plating baths.
Voltammetry is an extremely sensitive method (LODs in the µg/L range) for the analysis of electrochemically active substances such as inorganic or organic ions. Voltammetry is often used to determine transition metals such as nickel or cobalt in high purity water or wastewater. This technique combines a wide range of applications, short analysis times, and high precision with low costs of the required instrumentation.

ELECTROCHEMISTRY – SOLUTIONS FOR FUNDAMENTAL AND APPLIED RESEARCH

A wide range of electrochemical methods such as Cyclic Voltammetry (CV), time-resolved Chrono Methods (CM), and Electrochemical Impedance Spectroscopy (EIS) are suitable for fundamental and applied research in the field of new materials development and characterization for semiconductors.

NIR SPECTROSCOPY – MULTI-PARAMETER ANALYSIS WITHIN MINUTES

Near-infrared (NIR) spectroscopy can be used to assess the quality of acid etching baths. Compared to the traditional methods, results can be obtained within one minute and without the need for additional reagents or chemicals.

RAMAN SPECTROSCOPY – FAST, SIMPLE, REAL-TIME MONITORING

Raman spectroscopy is a non-destructive, real-time monitoring technique for cleaning solutions and etching baths. Raman signals are collected directly or even through tubing and windows with little or no sample handling. Surface-Enhanced Raman Spectroscopy (SERS) can be used to identify trace contaminants.

PROCESS ANALYSIS – DEPENDABLE ONLINE, INLINE, AND ATLINE SOLUTIONS

Process optimization through online monitoring of parameters such as water purity, acids in etching baths, and metals content in plating baths saves time and substantially lowers production costs. Metrohm Process Analytics offers several analytical techniques in different analyzer configurations for any need: titration, photometry, ion chromatography, NIR spectroscopy, XRF, and ion-selective measurements.
The same high quality Metrohm laboratory techniques from the laboratory can now be used right on the process line for the most accurate results delivered directly to the process control room.

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