



GLASS EXPANSION NEWSLETTER

Quality By Design

February 2004

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APPLICATION SPOTLIGHT

Ultra Trace Analyses for the Semi-Conductor Industry

INTRODUCTION

The miniaturization of semi-conductor devices requires a greater density of electrical components per chip. To accomplish this, engineers need a silicon wafer substrate and surface with as close to zero impurities as possible. For this reason, analysts in the semi-conductor industry must measure potential impurities in the silicon semi-conductor material as well as the reagents which come into contact with it.

SAMPLES

A number of sample types may be encountered in a semi-conductor quality control laboratory.

- * Bulk silicon dissolution: HF is used to dissolve silicon to determine its initial purity.
- * Wafer surface decomposition: HF is used to strip the top layers of silicon from the chip to measure the impurities which are removed.
- * Cleaning agent 1: A strong polar organic base such as TMAH (tetramethyl ammonium hydroxide) is used to dissolve leftover photoresist from the wafer surface.
- * Cleaning agent 2: Sulfuric acid (sometimes in combination with hydrogen peroxide) removes any remaining organic and inorganic residues.

The typical analytes of interest are the alkalis, alkaline earths, and transition metals. For the wafer surface analysis, sample volume is limited in order to minimize the dilution.

INSTRUMENTATION

Due to the low detection limits required, ICP-MS is the preferred analytical technique. The analysis of TMAH, in particular, because of its high carbon content, can result in

troublesome polyatomic isobaric interferences. Therefore, spectrometers with high resolution and/or a collision-reaction cell may provide an advantage. The "Cold Plasma" approach has also seen widespread use for these applications due to its ability to reduce troublesome background and interferences.

SAMPLE INTRODUCTION SYSTEM

The sample introduction system must be carefully selected to provide corrosion resistance and the lowest possible risk of sample contamination, while maintaining good sensitivity and stability so as to provide low detection limits.

Nebulizer - OpalMist



OpalMist Nebulizer

The OpalMist PFA nebulizer is recommended due to several characteristics as follows:

- * PFA used by Glass Expansion is of the highest purity, making it an ideal choice for this ultra trace application.
- * PFA is one of the most chemically inert materials known: one that can withstand reagents such as HF, sulfuric acid and hydrogen peroxide, as well as powerful organic and inorganic acids and bases.
- * The OpalMist is available in a variety of uptake rates, ranging from 50ul/min to 2 ml/min. The lower uptake rates are often selected when the application has limited sample volume. In addition, the fine mist produced by the high efficiency micro-flow (<0.2 ml/min) nebulizers reduces the production of oxides which might otherwise interfere.
- * The OpalMist is a self-aspirating concentric nebulizer, so that it can be used without a peristaltic pump to generate a fine and consistent aerosol. This has two benefits:
 - By removing the peristaltic pump from the system, a significant length of tubing is eliminated, dramatically speeding sample equilibration and flush, particularly when using lower uptake rates.
 - Perhaps more importantly, the elimination of the peristaltic pump means the elimination of the peristaltic

pump tubing which can be a source of significant contamination at these low levels.

Spray Chamber - HF Resistant Cinnabar Cyclonic



HF Spray Chamber

This Cinnabar cyclonic spray chamber is constructed of polypropylene and is recommended for the following reasons:

- * Its small (20ml) internal volume is ideally suited to the low uptake OpalMist nebulizer, establishing a homogeneous sample mist with minimal washout required.

- * The polypropylene material is of high purity. The internal surface of the spray chamber is pretreated in such a way as to facilitate fine mist production and to promote smooth draining. Special care is taken during the pretreatment process to eliminate the possibility of contamination of the chamber interior surface.

- * Unlike glass or quartz spray chambers, the polypropylene material is readily able to accommodate hydrofluoric acid matrices, in addition to other strong acids and various organics.

Torch - Semi Demountable or Fully Demountable Quartz Torch with Alumina Injector

A demountable torch is recommended so that the optimal sample injector can be installed. Alumina is not only available in high purity, but it also has the advantage that it is resistant to HF and other strong acids and bases and therefore makes a good choice for injector material. In the past, alumina injectors were not designed for ultra trace analyses. Low purity stock was used to save cost and machining tools were not appropriately chosen. Glass Expansion uses only the highest purity alumina and employs metal-free processes during the manufacturing cycle. Sapphire and platinum injectors are viable alternatives to alumina and are available for selected instrument makes.

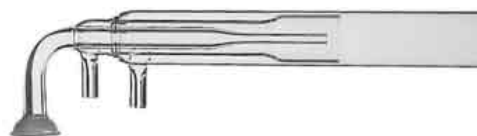
Sampler and Skimmer Cones - Platinum

For the same reasons of purity and chemical inertness, platinum-tipped cones are preferred. They provide generally cleaner backgrounds and greater lifetime. Also, since nickel is one of the transition metals typically being measured, it would be unwise to employ nickel cones in this application.

PRODUCT DESIGN FOCUS

The Fully Demountable Torch

The ICP torch comprises three circular concentric tubes – an outer tube, an inner or intermediate tube and an injector tube which delivers the sample to the plasma. The torch is usually made from quartz and the three tubes have traditionally been fused to form a single piece.



One-Piece Torch

While this approach has worked well, it does not offer the analyst any flexibility. Some samples require torch components with different dimensions or made of different materials. For example, quartz is attacked by HF, and samples containing HF require an injector tube made from a more inert material such as alumina. Also, organic solvents usually give best results when the sample flow is reduced and a narrow-bore injector is used. In order to accommodate the need for different injectors, the semi-demountable torch was introduced. With this design, the inner and outer tubes are fused to form a single-piece torch body while the injector is separate. The two parts are held in position in an adaptor or base with o-ring seals. The semi-demountable torch allows the injector to be conveniently changed in order to provide optimum performance with different sample types.



Semi-Demountable Torch

While the semi-demountable torch allows the injector to be changed, it does not address the common problem of outer tube damage. This tube is very close to the plasma and small changes in argon flow can cause it to overheat and melt. Also, salt particles from the sample can react with the quartz, causing devitrification. In either of these situations, the outer tube must be replaced. With a one-piece torch, this means that the whole torch must be replaced, even though the inner tube and injector may be fine. With a semi-demountable torch, the torch body must be replaced, even though the inner tube may be fine.

The solution to these problems is the fully demountable torch (FDT). With this design, the outer tube, inner tube

and injector are all separate components able to be replaced independently. With early FDT designs, the components were held in place by o-ring seals. However, it was very difficult to position the inner and outer tubes to the accuracy required using this method. It was only when Glass Expansion introduced the glass torch base that the tolerances required for routine replacement of all torch components could be met. With this design, the inner and outer tubes slide into a ground glass body that provides both the positioning and the gas seal. The glass body and the quartz tubes must be manufactured to micron-level tolerances so that any quartz tube can be fitted to any torch and still be perfectly positioned.



Fully Demountable Torch

The main benefits of the FDT are:

- * The outer, inner and injector tubes can be separately replaced so that the whole torch does not need to be discarded if one component deteriorates
- * Different injectors can be used for different samples eg. Ceramic injectors for HF solutions or narrow-bore injectors for organics
- * Ceramic outer and inner tubes are available for use when the silicon background needs to be reduced or to extend tube life when analyzing samples with high salt content

Glass Expansion manufactures fully demountable torches for Thermo, Varian, Jobin Yvon, Spectro and GBC models. Please send an email to enquiries@geicp.com for more information.

NEW PRODUCTS

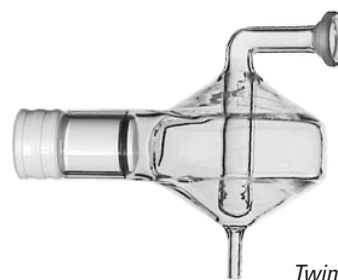
RF COILS FOR THERMO IRIS & PE OPTIMA 2000/4000 SERIES

Glass Expansion has expanded its range of RF coils to include coils for the Thermo Iris and the Perkin Elmer Optima 2000/4000 series. These coils are available with silver or gold plating, with silver giving the most efficient energy transfer but gold being more resistant to corrosion. The coil for the Thermo Iris also has a Teflon coating to help protect against corrosion. Each coil is supplied on a plastic former, ensuring correct dimensions are maintained during transport and simplifying installation. A re-usable

installation kit, which includes instructions on CD-ROM, is also available. Glass Expansion RF coils are available for Varian, Thermo and Perkin Elmer ICP models. Check our website at www.geicp.com to see the full range.

TWINNABAR SPRAY CHAMBER

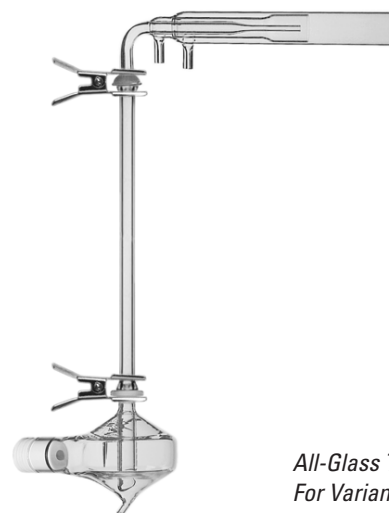
Glass Expansion is now offering the Twinnabar spray chamber for most ICP-AES and ICP-MS models. The Twinnabar is a combination of the highly successful Twister and Cinnabar spray chambers. The Twister spray chamber is a cyclonic spray chamber with a central baffle to filter the larger droplets. This design produces a finer aerosol, reducing the solvent load on the plasma and improving precision. The Cinnabar is a small-volume cyclonic spray chamber, designed for use with low-uptake nebulizers. The Twinnabar combines the benefits of these two designs to provide the best precision with low sample flow. The Twinnabar is available with the Helix o-ring free nebulizer fitting and a jacketed version is also available. Contact us at enquiries@geicp.com for information on a Twinnabar to suit your ICP.



Twinnabar Spray Chamber

ALL-GLASS TRANSFER SYSTEM FOR VARIAN VISTA

We have received a number of customer requests for an all-glass sample transfer system for the Varian Vista and this is now available for both the Axial and Radial configurations. This system eliminates kinked or twisted tubing, reduces condensation and improves sample transfer efficiency. It is also compatible with the Glass Expansion fully demountable torch. Send an email to enquiries@geicp.com for more information.



All-Glass Transfer System For Varian Vista

HINTS FOR THE OPERATOR

Care of Glass Expansion Nebulizers

Handling & Storage: Glass nebulizers should be treated with the same care that you would afford any delicate glass component. Glass can fracture and produce sharp, cutting edges, so handle all glass objects with slow, deliberate movements and don't apply large mechanical forces to them.

Take safe care of the nebulizer tips. Don't knock the tip of the nebulizer or leave it unprotected when not in use. Once a tip has been damaged, the nebulizer's performance cannot be restored. Teflon caps are provided with all Glass Expansion nebulizers. They should be used to protect the ends of the nebulizer when it is being stored. This prevents damage to the nebulizer tip, and prevents small particles from lodging in the bore.

Maintenance: Always start and finish each session by nebulizing a mildly acidic blank solution followed by demineralised water for a couple of minutes. This ensures that sample deposits or crystals don't form inside the nebulizer when the solvent dries out. Don't wash glass nebulizers in an ultrasonic bath since they can be easily damaged. However an ultrasonic bath can be used to clean OpalMist and PolyCon nebulizers.

If the sample capillary becomes blocked, use the Eluo nebulizer cleaner to remove the blockage. For the OpalMist, PolyCon and VeeSpray nebulizers, use the HF Eluo. For glass concentric nebulizers, use the standard Eluo. Regular use of the Eluo will help to ensure that the nebulizer remains clean and to maintain optimum performance.

Do not attempt to unblock nebulizers with wires or probes. This is very likely to result in damage to the nebulizer. If the nebulizer cannot be cleaned by using the Eluo or by soaking in a solvent, return it to Glass Expansion for cleaning.

Handy Tips: If you find that precision is getting worse, check the argon connection to your nebulizer. Tygon or other polymer tubes can harden over time and lose their flexible gas-tight grip. You should also check any connections in the nebulizer sample uptake tubing for possible air leaks.

Important Note: Hydrofluoric acid (HF) should not be used with glass or quartz. Using any amount of HF will damage the product. Glass Expansion nebulizers are supplied clean and ready to use. Do not pre-treat nebulizers with HF.

DISTRIBUTOR PROFILE

COURTAGE ANALYSES SERVICES - FRANCE

For more than 10 years Courtage Analyses Services (CAS) has produced and distributed technical consumables for CHNOS, EA-IR-MS and Proteins (Dumas). In France, North Africa, Benelux and in general French speaking countries, CAS also distribute a range of technical accessories for AAS and ICP including standard solutions, hollow cathode lamps, graphite tubes, nebulizers, torches, spray chambers and digesters. CAS supplies the highest quality products available and its team members, with their technical background, develop a close relationship with customers searching for performance and support. So the Glass Expansion innovative and high quality product range fits perfectly with the CAS market. CAS represents: Glass Expansion (Australia), Photron (Australia), Berghof (Germany), Environmental Express (USA), Analytika (Czech Republic), Altec (Czech Republic), Lemnatec (Germany), Birmingham Metal (UK), Santis (Switzerland), Brechbuehler (Switzerland), Id Analytical Services (France).

Contact Laurent Bertal or Sylvain Mallet at cas@onlinecas.com or check the website www.onlinecas.com.

INSTRUMENT NEWS

FROM AGILENT

Agilent Technologies have arranged a series of e-seminars on the latest applications developments of the 7500 Series ICP-MS. Hosted by ICP-MS experts; the 60-minute e-Seminars are a great way to gather valuable information from the comfort of your office or lab. The format is a slide presentation with separate audio via a phone line, so you will need a phone and a PC with internet access. You will also be able to post questions via a chat box which will be answered in real time. Participants can now phone toll-free to access the audio part of the presentation from the following countries: Austria, Belgium, Canada, Denmark, France, Germany, Ireland, Italy, Netherlands, Norway, Spain, Sweden, Switzerland, UK and USA. The e-Seminars: "Applying Reaction Cell ICP-MS to Routine Multielement Analysis" (Feb.25 - 2 pm EST), "Environmental Analysis: A Comparison of Cell and Non-cell ICP-MS" (Mar.16 - 9am EST, Mar.23 - 2 pm EST), "Developments in Speciation Measurement with ICP-MS as a Detector" (Mar.18 - 9am EST, Mar.25 - 2 pm EST), "Semiconductor Applications of Octopole Reaction Cell ICP-MS" (Mar.24 - 9am EST, Mar.31 - 2 pm EST), "Reaction Cell ICP-MS for Environmental

Metals Analysis" (Apr.8 - 11am EST). If you would like to sign up please visit the Agilent website at www.agilent.com/chem/icpms and follow the e-Seminar link.

FROM THERMO

Thermo Electron Corporation will launch a series of complete instrument packages to facilitate elemental speciation analyses at Pittcon 2004. These packages deliver "off the shelf" solutions for automated analysis in the routine and commercial laboratory environment. The new HPLC and GC coupling packs for the X Series ICP-MS can be easily integrated with other Thermo analytical tools such as HPLC (FinniganTM SurveyorTM or Spectra System) and GC (FinniganTM Focus or FinniganTM Trace) instrumentation. Interest in the use of hyphenated techniques for elemental speciation analysis in environmental, biological and clinical sample matrices is growing. Individual elemental species can exhibit various physical properties such as solubility, boiling point, toxicity and metabolic pathways in living organisms. With HPLC-ICP-MS and GC-ICP-MS, minimal sample preparation is required prior to analysis and rapid 'on-line' species separations are achievable. This ensures these techniques are ideally suited for the analyses of complex matrices with high species specificity and low detection limits. For more information about these new analytical solutions, contact analyze@thermo.com or visit the Thermo Electron booth # 1475 at Pittcon 2004, Chicago, 8 – 11 March.

GLASS EXPANSION NEWS

PITTCON 2004

A wide selection of Glass Expansion products will be on display at Booth #1834, Pittcon 2004, Chicago, March 8-11. The display will include nebulizers, spray chambers, torches, RF coils and accessories. Glass Expansion specialists will be on hand to answer your questions and assist you to choose the optimum components for your ICP.

WEBSITE

The Glass Expansion website at www.geicp.com is regularly updated to show the latest ICP components. You are reminded that you can access current prices and order online through our website.