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1. Instrument & Sample Preparation Environment

**Instrument Environment**
The ICP-MS instrument can perform low level (ppt) analysis in a variety of clean room environments. Class 1 and 10 clean rooms are the cleanest and least prone to environmental contamination, but they are also the most expensive to design, build, and operate. Class 100 to 10000 clean rooms can also be used for successful low-level analysis, but contamination incidents may be more frequent. By implementing some of the following methods, practices, and procedures in this document, it is possible to do very low-level semiconductor sample analysis even in a class 10000 clean room environment.

**Sample Preparation Environment**
The most important area that impacts the contamination of semiconductor samples is the sample preparation environment. Use of a laminar flow hood for standard and sample preparation can significantly help minimize the potential for contamination. When these hoods are purchased, the user can provide input into the design and configuration of the hood, controls, switches, power, etc. Following, are a number of considerations and suggestions in the design and operation of a laminar flow hood.

**Laminar Flow Hood Design**
- **Dimensions**: The dimensions of the clean room will determine to a certain extent the dimensions and number of laminar flow hoods that can be placed in it. The larger the hood work area, the better. A single hood work area dimension that has proven to work well is 91.5 cm (36") deep, 183 cm (72") wide, and 76 cm (30") high. All controls for power, lighting, DI water, power receptacles, etc. should be located on the front of the hood.
- **Hood construction material**: Laminar flow hoods are made either from a plastic coated metal or from 100 % polypropylene. The 100 % polypropylene is the preferred material for laminar hood construction.
- **Multiple workers** in the same hood are possible, although it can cause contamination problems as well as a safety hazard due to the nature of the chemicals used in the hood. Examples of tasks where you want only one person in the hood would be matrix elimination sample preparations, and surface metal extractions from silicon wafers. Multiple laminar flow hoods will allow multiple workers performing multiple tasks at the same time.
- **High efficiency particulate air (HEPA) filtration**: This air filtration system should remove 99.99 % of particles 0.6 um and larger. If possible, have the blower motor mounted in an exterior fashion to the hood. This will minimize hood vibration, which allows the use of balances in the hood.
- **Balances**: A balance is needed for formulating bulk chemical matrices in 100-600 gram quantities for matrix matching the calibration matrix to the sample matrix. The balance needs to be accurate only to two decimal places, and should have a high upper weight range of at least 500g.
- **Electrical requirements**: The analyst needs to examine what electrical devices will be used in the hood, and what their power requirements are. At least two, front mounted, 115 V, 60 Hz, 7.5 A duplex receptacles should be incorporated into the design. The need for a single
Lab Set-up

220 V duplex receptacle should be evaluated if spin dryers or spin coaters are going to be used in or near the hood. The hood work area should be well lit with a front mounted power control.

- **Sinks/drains:** One to two sinks are recommended depending on the specific facility and applications. The materials the drain is constructed from must be compatible with the chemicals that are disposed of in the sink.
- **DI or Ultra-Pure Water:** One source of DI water should be plumbed to each sink in the hood. The on/off control to the DI water should be on the front of the hood. Putting this control inside the hood requires the operator to lean inside the hood causing a safety and contamination hazard. A final stage polisher for Fab supplied water is recommended.
- **Vacuum:** Vacuum should be plumbed to the hood for wafer vacuum wand/pencil use if needed.
- **Nitrogen:** High purity nitrogen should be plumbed to the hood for cleaning and drying applications.
- **Chemical storage:** The appropriate chemical storage cabinets should be located close to the hood's working area. Some safety programs do not allow storage of acids in the shelving located in the lower part of the hood.

2. DI Water

Semiconductor fabrication facilities have "in house" ultra pure water (UPW) generation capabilities to support their specific manufacturing processes. They utilize large banks of ion exchange resin columns to clean or polish the feed water, and produce a very clean final product. The ion exchange columns are periodically replaced, and when they are, the metal/ion levels can fluctuate in the UPW for a short period of time. It is advisable to fit a high quality final polishing system in the laboratory to ensure a consistently pure water source.

3. High Purity Chemicals

High purity chemicals are required for most semiconductor analyses. A wide range of semiconductor grade reagents are available from suppliers such as the TAMA Chemical Company (http://www.tama-chem.co.jp/english/reag-contact.html), and Fischer Scientific i.e. Optima grade (https://www3.fishersci.com/main.jsp)

Chemical reagents with a guaranteed/certified metal impurity level below 10 ppt (pg/mL) are recommended, however, some chemicals are not available at this purity level. This grade of chemical is best suited for use in a class 100 clean environment or better. Care must be taken in the handling and use of these chemicals, since even in a class 1 environment they can be contaminated easily. Chemicals available at this purity grade include:
An alternative grade of chemical reagents have a guaranteed/certified metal impurity level below 100 ppt (pg/mL). Please note the wider variety of chemicals available at this purity level. These chemicals are recommended for use in a class 1000 clean environment or better. Please note: care must still be taken in the handling and use of these chemicals to prevent contamination. Common reagents available at this purity grade include:

<table>
<thead>
<tr>
<th>Chemical Name</th>
<th>% Composition</th>
<th>Chemical Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrofluoric Acid</td>
<td>38 %</td>
<td>500 grams</td>
</tr>
<tr>
<td>Hydrochloric Acid</td>
<td>20 %</td>
<td>500 grams</td>
</tr>
<tr>
<td>Nitric Acid</td>
<td>55 %</td>
<td>500 grams</td>
</tr>
<tr>
<td>Hydrogen Peroxide</td>
<td>35 %</td>
<td>500 grams</td>
</tr>
<tr>
<td>Ammonium Hydroxide</td>
<td>20 %</td>
<td>450 grams</td>
</tr>
<tr>
<td>Sulfuric Acid</td>
<td>98 %</td>
<td>250 grams</td>
</tr>
<tr>
<td>Perchloric Acid</td>
<td>70 %</td>
<td>500 grams</td>
</tr>
<tr>
<td>Hydrobromic Acid</td>
<td>47 %</td>
<td>500 grams</td>
</tr>
<tr>
<td>Acetic Acid</td>
<td>30 %</td>
<td>500 grams</td>
</tr>
</tbody>
</table>

Some chemicals suppliers also offer a water standard for those laboratories without a source of ultra-pure water (i.e. Tamapure-aa ultra-pure water). This standard is available in 500g quantities having metallic impurity levels guaranteed/certified to be less than 10 ppt (pg/mL).

4. High Purity Chemical Contamination Prevention

Contamination control for high purity reagents is essential for ensuring the integrity of the subsequent analysis. A chemical handling/storage technique that has worked well to prevent the contamination of high purity reagents in the clean room laboratory environment is outlined here.

1. Add 20 mL of an ultra-pure 1% nitric acid solution to a PTFE 125 mL container that has been cleaned according to the procedure described in Section 8, “Inert Lab-ware Cleaning”. Shake the container vigorously, and leave to settle for 10 minutes.
2. Go to the sensitivity tune screen in the Agilent ICP-MS ChemStation software, aspirate the solution and monitor the analytical masses associated with environmental contamination. These would include, but not be limited to, sodium 23, aluminum 27, magnesium 24, potassium 39, calcium 40, iron 56, and zinc 66. If the observed count rates are equivalent to those of the UPW alone, this would indicate no contamination in the cleaned bottle. If the count rates show that the vessel is contaminated, return it to the beginning of the cleaning cycle. It's worth noting that PTFE is the best material to work with when performing low-level analysis. PTFE is the easiest material to clean to low background levels both when new and when reused.

3. If the bottle is contaminant free, transfer the reagent to be used into the bottle. The 125 mL container will serve as a “secondary” reagent storage container. This will allow the original, or parent 500mL reagent container to be opened only 4 times in its useful lifetime, and thereby minimizing its probability of contamination.

4. If using Tama chemicals, please note that they will arrive with a set of instructions for minimizing contamination. The instructions are helpful with the following suggested modifications:

   - Each TAMA chemical comes in a PTFE container that is double bagged with one of the bags having the adhesive chemical label attached to it. The instructions suggest that the user reinsert the unlabeled bottle into the labeled bag each time it is used. It has been found that the reuse of the bag, which is often set on the working surface of the hood, can carry contamination to the container. It has been found that peeling the label off the bag and attaching it to the Teflon container is a better practice.

   - Each TAMA chemical comes in a PTFE container that has an additional PTFE insert in the neck of the bottle. These inserts should be disposed of when the container is first opened, and not replaced to the neck of the container since it also acts as a mechanism of contamination transport to the reagent container.

5. Suppliers of Instrument Calibration Standards

There are a number of companies that can provide high quality instrument calibration standards suitable for use in semiconductor analysis. A partial list includes:

Inorganic Ventures, Inc.
195 Lehigh Ave, Suite 4
Lakewood, NJ 08701
Tel: 1-800-669-679 or 908-901-1900
Fax: 908-901-1903
6. Pipettes

Laboratories should have a variety of pipettes to dispense volumes from 20 uL to 1000 uL. These can be found in virtually any laboratory supplies catalog.

7. Inert Lab-Ware

Inert lab-ware is manufactured from a number of different materials, and is available from a number of different manufacturers. Generally, there are two concentration ranges for semiconductor type samples, the ug/L (ppb) range, and the ng/L (ppt) range. For the ppb sample analysis range, inert lab-ware can be made from polypropylene, polymethylpentene, and polyethylene. For the ppt sample analysis range, inert lab-ware should be made from PTFE, either FEP or PFA. Experience has shown that volumetric type lab-ware is not very useful in the semiconductor laboratory since most formulating work is done by weight. Wide mouth bottles work the best when working by weight. To arrive at a more exact weight, a PTFE squirt bottle filled with the matrix being weighed out provides very accurate dispensing.

8. Inert Lab-Ware Cleaning Procedure

Contamination control is critical in semiconductor sample analysis. Good lab-ware cleaning methods and laboratory technique can significantly reduce the occurrence of contamination. For this reason, all materials and containers that come into contact with the samples should be cleaned. A general rule is to clean with the same acid/acids that will be analyzed on the ICP-MS, or with the industry standard cleaning solution (SC1). For example, if a sulfuric acid manufacturer wanted to clean their inert lab-ware, they would use a 5% sulfuric acid cleaning solution to do so. Most semiconductor facilities analyze almost all of the manufacturing acids, and find it useful to use this cleaning procedure for containers and miscellaneous lab-ware.

Note: The gloves worn by the analyst that should always be rinsed with UPW before working with samples, calibration standards, or performing this cleaning procedure.
Container Cleaning Procedure
This cleaning procedure is intended for closed vessel type containers. The cleaning solution is a 3% by wt mixture of HF, HNO₃, H₂O₂, and UPW. Other cleaning solutions can be used that suit the individual laboratory’s needs. Suggestions for other cleaning solutions are SC1 (7% H₂O₂, 7% NH₄OH, 86 % UPW), or a two-step leach that uses nitric in the first step, and hydrochloric in the second. For ppb level semiconductor samples, a cleaning solution of 5 % nitric acid can usually be used.

1. Rinse the container 3 times with UPW and pour contents to waste.
2. Prepare the necessary volume of the cleaning solution to fill all of the containers to be cleaned.
3. Fill all of the containers with the cleaning solution and replace the cover or cap.
4. Let the container leach for one week, then invert the container and allow leaching for another week. If there is a pressure buildup during the leach cycle, release the pressure and continue with the procedure.
5. At the end of the cleaning cycle, pour the cleaning solution to waste.
6. Rinse the container out three times with UPW. Store the cleaned container with either UPW or 1% nitric acid.
7. Before using the container, rinse it three times with the chemical matrix the container will be used with.

Miscellaneous Lab-Ware Cleaning Procedure
This cleaning procedure is intended for smaller objects that are used in the laboratory such as pipette tips, test tubes, Teflon pipetting cups, etc.

1. Place all small objects to be cleaned in the appropriately sized leach tank containing the cleaning solution.
2. These should soak in the cleaning solution for 1-2 weeks.
3. Before these items are used, they should be rinsed (3 times) with UPW and then be rinsed (3 times) with the chemical matrix they will be used with. After these cleaning/rinsing steps, the item is ready for use.

9. Instrument Cleaning Procedure
Before low level analysis (ppt) analysis can be performed on an ICP-MS system, the sample introduction system must be cleaned so that the backgrounds can be lowered, and contamination problems can be eliminated.

- All quartz ware should be cleaned in 5 % nitric acid overnight. Polypropylene and Teflon can be cleaned in more aggressive cleaning solutions such as HF and hydrogen peroxide. Do not clean any glassware or quartz ware in solutions containing HF.
- The sample and skimmer cones should also be cleaned in 5 % HNO₃. Do not expose the cones to prolonged nitric acid exposure.
- Aspirating a cleaning solution appropriate to the specific application should clean the nebulizer. For example, if performing an analysis that uses HF, use a dilute solution of HF and HNO₃. A sulfuric manufacturer would use a dilute sulfuric acid to clean the nebulizer.
10. Miscellaneous Items

PTFE sample tubing

- 0.3 mm ID, Agilent part # G1820-65478. This tubing can be pumped, or used for self-aspiration at a sample uptake rate of 200-300 uL/min.
- 0.2 mm ID, Agilent part # G1820-65227. This tubing can be pumped, or used for self-aspiration at a sample uptake rate of 50-150 uL/min.
- 0.15 mm ID, Agilent part # G1820-65227. This tubing can be pumped, or used for self-aspiration at a sample uptake rate of 50 uL/min.

Inert lab-ware cleaning tank
Polypropylene cleaning tanks can be purchased from most laboratory supply catalogs. They should be cleaned thoroughly before they are filled with a cleaning solution. Since these usually reside in the clean room hood, be careful not to buy a size that is too large. Hood space can be optimized if a stacking variety is used.

Peristaltic pump tubing
A variety of sizes are recommended.

Stainless steel cutting tool
The ideal tool for cutting sample introduction tubing is one made from stainless steel.

Small storage containers
These are used to organize/store ICP-MS parts, components, different sample introduction systems, etc.