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# Analysis of Organic Contamination In Semiconductor Processing

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The cleanroom ambient contains numerous sources of volatile organic contaminants which can deposit on wafers. The effects of these contaminants on various aspects of device processing are reviewed. Possible sources of the contaminants, along with analytical techniques that can be used to identify and measure organic compounds are described.

## INTRODUCTION

"Water insoluble organic compounds tend to make semiconductor and oxide surfaces hydrophobic, thus preventing the effective removal of adsorbed ionic or metallic species... Typical molecular contaminants... include... greasy films that are deposited when surfaces are exposed to room air or stored in plastic containers" (1). In their 1970 paper introducing the RCA clean, Kern and Puotinen identified two of the main features of organic contamination: airborne contamination can be deposited from processing ambients, and the effects of organic contamination can be magnified by their influence on the effectiveness of inorganic cleans. In this paper, we will review recent studies of the effects of organic contamination on device processing and briefly discuss some sources of the contaminants. We will also give overviews of cleaning methods being used to remove organics and of analytical techniques used to characterize molecular contaminants.

## EFFECTS OF ORGANIC CONTAMINATION ON DEVICES

### Oxide Quality

Kasi, *et al.*, showed, by exposing cleaned wafer surfaces to controlled amounts of known organic molecules, that significant amounts (10E15 atoms/cm$^2$) of hydrocarbon can cause serious degradation of metal-oxide-semiconductor (MOS) devices grown on hydrogen passivated, HF cleaned, (100) Si (2). Measurements of the amount of bulk charge in the oxide and of the number of defect sites at the Si-$SiO_2$ interface indicated that carbon was distributed throughout the oxide. Organic contaminants that had high sticking probabilities, desorption temperatures higher than the temperature where hydrogen passivation is lost, and that fragment easily during thermal processing had stronger effects on performance. The amount of organic material on the wafer surface was determined from the area under the carbon 1s peak measured by X-ray Photoelectron Spectroscopy (XPS). The C, Si, and O XPS peaks gave information on the bonding between carbon and Si.

Differences in the behavior of gate oxides were observed by Iwamoto and co-workers to depend on whether a wafer was processed with or without exposure to the clean room ambient (3). Attenuated Total Reflection Fourier Transform Infrared Spectroscopy (FTIR-ATR) measurements showed that the amount of contamination on the oxide increased with exposure time in the clean room. After deposition of a phosphorus-doped polysilicon, carbon was found by SIMS to be present at the polysilicon/ $SiO_2$ interface.

Saga and Hattori eliminated the deleterious effects of contamination on oxide quality by a suitable choice of ambients as the wafers were being loaded into the oxidation furnace (4). Electrical measurements detected degradation of gate oxide integrity if wafers were intentionally contaminated with the anti-oxidant butylated hydroxytoluene (BHT) and loaded into the gate oxidation furnace in the presence of a nitrogen atmosphere; SIMS detected carbon in the silicon dioxide. Addition of a small amount of oxygen to the nitrogen caused degradation and evaporation of the BHT, as seen by improved electrical parameters, and lower levels of carbon in the $SiO_2$.

### Oxide Growth Rates

Licciardello and co-workers found that the initial room temperature oxide growth rate of HF-etched (hydrophobic) Si wafers was retarded by the presence of surface organics (5). The growth kinetics were consistent with an oxidation which takes place by the diffusion of

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oxygen through an overlayer on the silicon surface. Hossain, *et al.*, showed that high temperature oxide growth on hydrophobic wafers was affected by a layer of contamination; while no effect was found for hydrophilic wafers (6).

## CVD of Silicon Nitride

As discussed above, organic contamination affects processing in non-oxidizing, ambient atmospheres. Saga and Hattori exposed wafers to the clean room atmosphere or stored the wafers in plastic boxes and found that initial nitride growth in a low-pressure chemical vapor deposition (CVD) was retarded (7). After exposure times of one month, an incubation time of several minutes was observed for CVD nitride growth, while clean wafers showed no incubation time. Thermal desorption gas chromatography mass spectrometry (GC-MS) found BHT, associated with antioxidants, and dibutylphthalate (DBP), a plasticizer, on wafers stored in a plastic box. DBP, tributyl tricarboxylate, and dioctylphthalate (DOP), also a plasticizer, were found on wafers exposed to the clean room air. The wafers exposed to the clean room contained higher levels of contamination than those stored in boxes.

## Silicon Epitaxial Growth

Rapid thermal chemical vapor deposition (RTCVD) combines rapid thermal annealing and CVD to produce a thin, high quality silicon epitaxial layer with abrupt profiles. Kim, *et al.*, etched wafers in HF, and, after a $H_2$ bake at 1000 C for 45 seconds to remove the native oxide, grew a RTCVD epitaxial layer at 1000 C (8). They identified beta SiC precipitates at the substrate-epi interface by SIMS and transmission electron microscopy. By growing successive layers, and by only observing the precipitates at the interface between the substrate and the first epitaxial layer, they showed that the carbon originated at the initial surface, not in the process gas or from the vacuum system.

## Wafer Doping

Clean room materials have caused unintentional doping of Si wafers by both boron and phosphorus. Stevie, *et al.*, improved the quantification of small amounts of boron in Si by SIMS by creating a sample where the matrix was identical on both sides of the interface, eliminating the problem of equilibrium of the primary beam species and secondary ion yield in the region before the implant range of the analysis beam is reached (9). A layer, such as polysilicon, was deposited on Si, the layer was then exposed to atmosphere and then covered with a layer identical to the first layer. After exposure to the clean room air, boron was found on the wafer. The source of the contamination was borosilicate glass, which was present as sub-micron glass fibers in HEPA filters. Electron microprobe and inductively coupled plasma atmospheric analyses found that boron could constitute 10-20% of the weight of HEPA filters. Lebens and co-workers found that an organophosphate flame retardant in polyurethane potting material used to secure and seal filter material to the module in some HEPA filters caused phosphorus doping (10).

## Wafer Bonding

Si wafer bonding, one method for producing silicon-on-insulator (SOI) structures, requires homogeneous bonding across the joined surfaces. Unbonded areas, or bubbles, were shown by Mitani, *et al.*, to be due to hydrocarbon contamination originating in storage in plastic boxes and from exposure to controlled amounts of organic vapors (11).

## Effect on Photoresists

Several groups have shown that photoresists are affected by various organic contaminants, particularly organic amines (12, 13).

# SOURCES OF ORGANIC CONTAMINATION

Clean rooms have been designed to remove particulate contamination, with less thought given to the outgassing properties of the materials used to construct the rooms. Wall materials, window materials, floor and ceiling materials, and air filters all have organic components. Delivery systems for liquids and gases, bottles, clean room garments, and especially the plastic boxes used to hold wafers are all potential sources of volatile contaminants.

Evaluation of the contamination potential of materials can be carried out by measuring the outgassing of the material itself or by measuring the amount of contamination deposited on a wafer surface after exposure to the material. The importance of measuring the wafer surface was demonstrated by Saga and Hattori in their analysis of the effects of storage in plastic boxes (14). Although unpolymerized monomers and oligomers outgassed in large quantities, the major species found on the wafer surfaces originated from the small quantity of additives in the materials, such as antioxidants, plasticizers, and cross-linking agents. In general, the lower the vapor pressure and the lower the molecular

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weight the more easily the materials adsorbed onto the wafer surface. The material's polarity also affected its adsorption.

## PROCESSING TO MINIMIZE EFFECTS OF ORGANIC CONTAMINATION

*In situ* processing, in which the wafer surface is not exposed to the clean room atmosphere between cleaning and processing, and cleaning procedures using ultraviolet light (UV), ozone, and, more recently, high temperature water have been developed to supplement the traditional cleaning procedures, such as the RCA clean. The desire to reduce the large volume of contaminated liquid waste from the cleaning processes is another incentive to develop alternate cleaning procedures.

Vig reviewed the use of UV/ozone cleaning procedures to remove organic contamination (15). The removal mechanism was seen to be primarily a photosensitized oxidation process. Short wavelength UV light excites and/or dissociates the contaminant molecules and dissociates molecular oxygen and ozone to atomic oxygen. The excited contaminant molecules and the free radicals produced by the dissociation of the contaminants react with atomic oxygen to form simpler volatile molecules, such as $CO_2$, $H_2O$, $N_2$, *etc.* The rate of contaminant removal for the UV/ozone combination was greater than two orders of magnitude faster than when either was used alone.

Hydrocarbon removal by a UV/$O_2$ combination was studied by Kasi and Liehr by exposing wafers to known gases (16). After HF etching to produce a hydrogen-terminated, hydrophobic surface, wafers were contaminated with a drop of a specific hydrocarbon, spin-dried in a nitrogen atmosphere, and moved without exposure to air through a vacuum load lock for *in situ* cleaning and analysis. The rate of hydrocarbon removal in $O_2$ gas in the presence of UV depended on the nature of the deposited hydrocarbon.

Ohmi showed that ozone-injected ultrapure water was effective in removing organic impurities and promoting native oxide growth on Si (17). Bakker and Hess have developed a cleaning process based on water and water/$CO_2$ mixtures at elevated temperatures (100 to 700 C) (18). Below 100 C, water dissolves inorganic (ionic) species. Between 100 C and the critical point (374.4 C) water's properties change from polar to nonpolar. At the critical point, hydrocarbons become very soluble, while ionic salts no longer dissolve. The cleaning properties of water can thus be adjusted by controlling its temperature. Bakker and Hess also describe how water at high temperature can dissolve $SiO_2$, thus removing any organic contaminants in native oxide films.

## ANALYTICAL TECHNIQUES FOR ORGANIC CONTAMINATION

In selecting the analytical techniques to be covered in this review, two basic criteria have been used. First, the relevant techniques can be used to analyze contamination that deposits on wafers, *i.e.*, methods used only for air sampling are not discussed. Second, appropriate techniques for evaluating organic contamination must provide information that allows individual organic compounds or classes of compounds to be identified. In other words, these techniques must provide either direct or indirect chemical identification and/or bonding information.

The review of the known effects of organic contamination presented earlier highlights the importance of both of these points. Saga and Hattori noted significant differences between the outgassing products from plastic boxes and the compounds that ultimately deposit on wafers (14). At the same time, identifying a contaminant as simply containing certain elements is not sufficient, because specific compounds or classes of compounds will adhere to surfaces preferentially and will produce different effects depending on subsequent processing. In addition, more specific information can be valuable for tracing contaminants to their sources in order to reduce or eliminate them.

There are a number of analytical techniques that can meet these requirements, ranging from traditional wet chemical analysis to advanced surface analysis. However, the techniques most commonly used to evaluate organic contamination on semiconductor surfaces are Gas Chromatography-Mass Spectrometry (GC-MS), Ion Mobility Spectrometry (IMS), Time-of-flight Secondary Ion Mass Spectrometry (TOF-SIMS), X-ray Photoelectron Spectroscopy (XPS; also known as Electron Spectroscopy for Chemical Analysis or ESCA), and Fourier Transform Infrared Spectroscopy (FTIR).

These techniques can be classified into two groups. The first, covering GC-MS and IMS, includes methods that detect contaminants after desorption from a surface. In the second group, which includes TOF-SIMS, XPS and FTIR, the surface of the wafer is analyzed directly. Each of these techniques is discussed below, and a summary of some advantages and limitations for each is in Table 1.

### Desorption techniques

Many common types of organic contamination are relatively volatile materials that deposit on wafer surfaces from the air. IMS and GC-MS, the two main techniques that rely on desorption of contaminants, take advantage of this volatility by measuring material outgassing from a wafer under controlled conditions. For both techniques, the desorption is typically induced thermally.

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**TABLE 1.** Summary of Techniques Used to Analyze Molecular Contaminants on Wafers

| Technique | Advantages | Limitations |
|---|---|---|
| GC-MS | -Good sensitivity for volatile contaminants<br>-Direct chemical ID possible (MS)<br>-Separates contaminants before identification<br>-Semi-quantitative results can be obtained | -Poor sensitivity for ionic, nonvolatile or thermally unstable contaminants<br>-Long analysis times<br>-Detection limits poorer than IMS |
| IMS | -Excellent sensitivity for volatile contaminants<br>-Separates contaminants<br>-Quantification is possible | -Poor sensitivity for ionic, nonvolatile or thermally unstable contaminants<br>-Sensitive to overloading<br>-No direct chemical information |
| TOF-SIMS | -Surface of wafer analyzed directly<br>-Excellent sensitivity for nonvolatile/ ionic contaminants<br>-High resolution mass spectrum for chemical ID | -Vacuum technique; requires cold stage to detect volatile contaminants<br>-Absolute quantification is difficult because it requires standards |
| ESCA/XPS | -Surface of wafer analyzed directly<br>-Quantitative for elements and bonding states | -Not as sensitive to low levels of contamination<br>-Vacuum technique; requires cold stage to detect volatile contaminants<br>-Chemical identification ability is limited |
| FTIR | -Surface of wafer analyzed directly<br>-Atmospheric pressure technique, for both volatile and nonvolatile contaminants<br>-Extensive libraries available to ID contaminants | -Not as sensitive to low levels of contamination<br>-No separation of contaminants, which complicates data interpretation |

IMS has the advantage of very high sensitivity, which makes it possible to detect the small amounts of material that can affect device performance. However, this high sensitivity also makes IMS susceptible to overloading by more abundant, but relatively harmless species. GC-MS is not as susceptible to overloading, but special procedures may be required to detect low levels of contaminants. A practical advantage of GC-MS is its presence in many labs.

### *Gas Chromatography-Mass Spectrometry*

GC-MS combines the separation capabilities of a gas chromatograph with the chemical identification abilities of a mass spectrometer. To evaluate contaminants present on wafers, GC-MS is typically combined with some type of thermal desorption apparatus that is used to volatilize the contaminants. This combined technique is also referred to as Thermal Desorption GC-MS or TD-GC-MS.

While commercially available TD-GC-MS instruments designed specifically for use with wafers are now available, many laboratories have developed their own in-house apparatus that can be attached to available GC-MS instruments. The time, temperature and other desorption conditions can be varied in the desorption step, though some efforts are being made to standardize protocols for specific types of analyses (19).

After the contaminants are desorbed thermally into a carrier gas, they are typically trapped on a cold finger before entering the GC. When the thermal cycle is completed, the cold finger is heated quickly to re-volatilize the contaminants. An inert carrier gas is used to take the contaminant mixture through the column, where the various compounds are partitioned into separate components based on their interaction with the stationary phase of the column. At the end of the column, each component is detected and tracked as a function of time.

In GC-MS, the gas chromatographic separation, which separates chemical species but does not provide chemical information, is followed by a mass spectrometric analysis. The mass spectrum obtained for each component separated by the GC provides direct chemical information that often can be used to identify the species. In practice, analysis of all components can be a time-consuming process, so the species reported for a given analysis may be limited to those above a selected minimum peak intensity or to specific compounds that are of interest in the analysis. A combination of standards, available reference compounds, mass spectrometry and, less frequently, retention times, is used to identify and semiquantitatively report data for most species observed in the analysis.

GC-MS has several advantages in analyzing organic contamination. It is an atmospheric technique, so no species are lost due to vacuum conditions. The use of standards and reference materials make it possible to get

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semiquantitative results fairly easily. As noted above, instruments are also available in many laboratories.

Some limitations of the technique are its detection sensitivity, which may require special procedures to detect low levels of contaminants; analysis times, which can be fairly long; possible cross-contamination that may require purging after very dirty samples; and insensitivity to non-volatile or easily condensable contaminants.

### *Ion Mobility Spectrometry*

IMS separates ions based on their differences in travel time in an applied electric field. A silicon wafer is typically placed in a quartz sample chamber. The temperature of the sample is increased, and the volatile components are vaporized and mixed with a heated carrier gas, such as nitrogen. The carrier gas and organic volatiles are carried into a reaction chamber, where a foil of $^{63}Ni$ emits 60 keV beta rays and ionizes the gas. As the gas is carried through the reaction chamber, sample molecules are ionized through a series of charge transfer and energy transfer reactions between the carrier ions, the electronically excited species, and neutral sample molecules (20).

An applied electric field moves the ions through the reaction region until they reach a shutter grid. The grid is opened repeatedly to admit pulses of ions into a drift region. Separation of the ions occurs in the drift region due to the differences in mobility of the various ions in the presence of the inert carrier gas and the applied electric field. In general, smaller, lighter ions have higher mobilities.

Under normal operating conditions, ions migrate at velocities between 1 and 10 m/s, with arrival times on the order of 3-30 ms. After monitoring the collector current for approximately 30 ms, the grid is reopened and the measurement repeated to improve the signal to noise ratio.

IMS peaks are typically broad compared with the range of possible drift times, and identification of unknown species by mobility data alone is difficult. In practice, species are usually identified using a combination of standards and supplemental mass spectrometric analysis.

IMS has several significant advantages in measuring the small quantities of organic contamination that can affect device performance. Since it can measure ion currents below $10^{-12}$, IMS has very high sensitivity; picograms ($10^{-12}$) and even femtograms ($10^{-15}$) of contaminants have been detected. As noted for GC-MS, IMS is an atmospheric technique so no species are lost due to vacuum conditions. By collecting mobility data as a function of sample temperature, additional separation data can be obtained.

The sensitivity of the system to overloading, and the possibly long cleaning periods required to purge the system preclude characterizing the outgassing properties of polymers or highly outgassing samples directly. As noted for GC-MS, the technique is also insensitive to contaminants that are nonvolatile or have only low volatility. Extreme care is even needed in handling silicon wafers to avoid introducing extraneous contamination. Budde, *et al.*, provide a good review of the application of IMS to device analyses (21).

## Surface Analysis Techniques

Unlike the desorption methods discussed above, surface analysis techniques are used to analyze the wafer surface directly. The advantage of this approach is a higher sensitivity to less volatile polar or high molecular weight contaminants. This can also be important when reactions occur on a surface, for example when salts form from volatile organic amines and anions such as sulfates. A disadvantage of direct analysis is that there is no separation step, so spectra obtained from the wafer contain data from all of the compounds detected.

### *Time-of-flight Secondary Ion Mass Spectrometry*

In TOF-SIMS, mass spectra are obtained by detecting positive or negative ions generated from a surface. A wafer or piece of wafer is placed in a sample chamber that is typically at a pressure of $10^{-8}$ to $10^{-10}$ torr (ultrahigh vacuum, UHV, or near-UHV). A pulsed primary ion source, for example $^{69}Ga^{+}$, $Cs^{+}$ or $Ar^{+}$, is used to bombard the surface of the sample, causing the emission of molecular and atomic secondary ions. The secondary ions are electrostatically accelerated into a field-free drift region. At the end of the drift region, the mass of each ion is determined from its flight time (22).

Secondary ions are generated and recorded with each primary ion pulse. A complete spectrum is generated by accumulating secondary ions detected in successive pulses for extended periods, typically 1-15 minutes. Typical analytical areas for the detection of organic contamination are on the order of 80 μm x 80 μm.

There are several advantages of TOF-SIMS for the measurement of organic contamination. It has very good sensitivity for low levels of contaminants and for low volatility or thermally unstable contaminants that can be difficult to analyze by desorption techniques. The high mass resolution of the mass spectrometer can provide unambiguous identification of chemical species. In addition, because the analytical areas are localized and the ions detected are coming from specific, identifiable regions of a wafer, it is possible to compare the levels of contamination from different regions on the same wafer (23).

The most important limitations of the technique are related to difficulties with quantification and to the effects of the UHV conditions of the experiment. Quantification

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is complicated by the large differences in relative ion yields among organic ions. This means that, at present, it is only possible to make relative comparisons of the intensities of a particular species among a series of wafers. The standard UHV conditions of the TOF-SIMS analysis mean that more volatile compounds present on wafers are often removed from the sample before it is analyzed. This problem can be mitigated by using a cold stage to perform the analysis (24).

### *X-ray Photoelectron Spectroscopy*

In XPS, also known as Electron Spectroscopy for Chemical Analysis (ESCA), X-rays excite photoelectrons from a sample surface. The analysis is performed by placing a wafer or piece of wafer in a UHV sample chamber. Soft X-rays, usually either Al-kα or Mg-kα, excite photoelectrons in the near surface region (10-100 Å) of a sample. The photoelectrons are collected by an electrostatic lens and then are passed through a high resolution analyzer before detection. The photoelectron binding energy is characteristic of the element and chemical bonding environment from which the photoelectron originated. Information about oxidation states is obtained by comparing lineshapes and peak positions with published literature values. Quantitation is performed using sensitivity factors (25).

The advantages of XPS for the analysis of organic contamination lie in its quantitative capabilities and its ability to detect compounds that are nonvolatile or have low volatility. It is also a valuable tool for measuring the amount and type of carbon present after subsequent processing or when species such as silicon carbides may be present (2).

The limitations of the technique for organic applications are related to the relatively low sensitivity of the method and the limited chemical identification that is possible. Typical detection limits are on the order of 0.01 atomic percent, significantly worse than those obtainable with TOF-SIMS or IMS. The chemical state identification possible with XPS provides general information about the type of bonding present, but it is generally not possible to identify specific organic contaminants from XPS data. Finally, as with TOF-SIMS, the UHV analysis conditions can introduce problems in the analysis of volatile contaminants.

### *Fourier Transform Infrared Spectrometry*

In FTIR analyses, the absorption of infrared radiation by a sample is measured. The IR radiation is absorbed at wavelengths corresponding to natural vibrational frequencies of covalent (molecular) bonds. The portion of the infrared spectrum that is of interest lies between 4000 and 400 $cm^{-1}$. In Fourier Transform spectrometers, analyses are performed using a radiation source that emits an infrared beam covering all of these wavelengths simultaneously. The waveform that is measured after interaction with the sample is deconvoluted to produce an infrared spectrum. Spectra are typically presented as either absorption or percent transmittance versus infrared frequency.

For wafer contamination analysis, samples generally must be analyzed using either Attenuated Total Reflectance (ATR) or grazing angle adaptations of the technique that enhance the surface sensitivity of the measurement.

Since most organic compounds contain several types of covalent bonds that absorb different frequencies of IR radiation, the complete spectrum for individual compounds can provide a relatively unique fingerprint for the material. Large databases of IR spectra are available that can make identification of unknown materials very practical.

The major advantage of FTIR in the analysis of organic contamination is this ability to make use of extensive databases to identify compounds. Another advantage of the technique is that it can be used to look directly at wafer surfaces without requiring vacuum conditions. This minimizes difficulties in the analysis of volatile contaminants.

The chief disadvantage of FTIR is its relatively poor detection sensitivity. This is especially a problem when a contaminant needs to be identified at low concentrations. A second disadvantage is the difficulty of identifying a particular contaminant when, as is often the case, more than one compound is present on the sample. As noted in the general discussion of the direct surface analysis techniques, these methods do not separate the contaminants prior to identification. For TOF-SIMS, the high mass resolution and specificity of individual mass spectral peaks minimizes the difficulties caused by this, but it is a problem for FTIR.

## Summary of Analytical Techniques

The analytical techniques discussed above all meet the two criteria that were previously defined. Each can be used to analyze contaminants that deposit on wafers, and each provides molecular or bonding information, either directly or indirectly.

In the explanations of each technique, other factors are discussed that can be important in determining the correct tool for a particular analysis. These include: (a) detection sensitivity, since levels of individual contaminants are generally very low; (b) consideration of the volatility and surface affinity of the contaminants; (c) the type of chemical information provided by the technique; (d) the ability to provide quantitative or semi-quantitative results; (e) an awareness that contamination is often chemically altered and redistributed throughout a



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material during subsequent processing. Beyond these factors, others, such as availability and cost, are often important in the selection of a technique for a particular application.

In general, no single analytical technique can provide the answer to every contamination problem. The specific requirements of individual situations will often dictate the choice of technique that is most appropriate. In the analysis of organic contamination, this choice can be complicated because the effects of different contaminants are only beginning to be understood.

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