

SIMS Solutions for Next Generation IC Processes and Devices

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Introduction

Secondary Ion Mass Spectrometry (SIMS) can be a powerful tool for trace element failure analysis investigations. Dynamic SIMS is a depth profiling technique that features parts-per-billion (ppb) detection limits for many elements and at least parts-per-million (ppm) detection limits for every element in the periodic table including hydrogen. Dynamic SIMS can be used to detect contamination distributed throughout films or at interfaces. Dopant profiling is a common application and stoichiometry can also be measured for some materials systems.

Dynamic SIMS cannot directly make measurements on deep sub-micron devices. Lateral analysis dimensions are typically $800 \mu\text{m}^2$ to $100 \mu\text{m}^2$. With special effort, $10 \mu\text{m}^2$ areas can be depth profiled, a much larger area than that of a single sub-micron device. Special test structures are often incorporated into wafer patterns in order to accommodate SIMS measurements. Shrinking lateral dimensions have been accompanied by ultra-thin layer structures. Recent advances in SIMS depth profiling have made it possible to resolve structures as thin as 2 nm thick.

With excellent detection sensitivity for all elements, SIMS can be used to detect contaminants that may be interfering with device performance, dopants that may not have intended concentrations, or dopants that may have diffused into undesirable locations. Layer thickness can be measured directly because SIMS is a depth profiling technique. Stoichiometry can be measured for some materials systems and compared with expectations.

Failure analysis using SIMS often takes the form of a “good vs. bad” study. SIMS, with its tremendous sensitivity, will frequently find contamination through films and at interfaces in both “good” and

“bad” devices. Some contaminants may be irrelevant to device failure. Dopant levels can be measured, but cannot independently determine if the dopant level causing a problem. The “good vs. bad” comparison is necessary in order to determine the significance of the findings.

A successful outcome to SIMS failure analysis experiments is a product of good experimental design and planning. In this article, we outline the considerations that are part of the experiment planning process so that the chances of solving problems can be maximized.

SIMS Basics

In a SIMS analytical system, ions are extracted from an ion source, and then accelerated and focused down a column in order to form an ion beam. The ion beam is then directed onto a sample surface in a square raster pattern that results in sputter removal of the sample surface. During the sputtering process, some of the sample material is ionized. This ionized sample material is accelerated by an electric field away from the sample forming a second (or secondary) ion beam. The secondary ion beam is passed through a mass spectrometer that identifies the mass of the ionized particles. The mass filtered secondary ion beam is then directed to a detector that measures the intensity of the secondary beam. By careful calibration to standards, the secondary ion intensities are quantified.

Secondary Ion Generation

The SIMS process of sputtering a sample surface is a very inefficient generator of secondary ions. Only a few percent of the sputtered sample material is ionized. Ionization probabilities can be increased through the use of reactive primary ion beams. An oxygen primary ion beam can be used and will increase the probability of positive secondary ion formation. Alternatively, a cesium

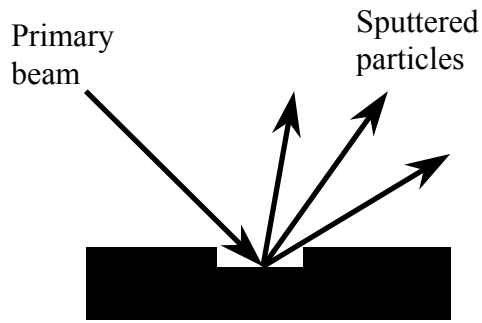


Figure 1: A cross-section of a sample showing the SIMS sputtering process and crater formation.

primary ion beam will increase the probability of negative secondary ion formation. The choice of primary beam is therefore often determined by the elements that are to be analyzed.

Vacuum Compatibility

SIMS primary and secondary ion beams need to travel through vacuum in order to have a mean free path that is long enough to reach the intended destinations. Therefore samples need to be vacuum compatible.

Depth Profiles

The SIMS sputtering process etches a crater into the sample surface (Figure 1). Secondary ions are collected continuously as the primary ion beam

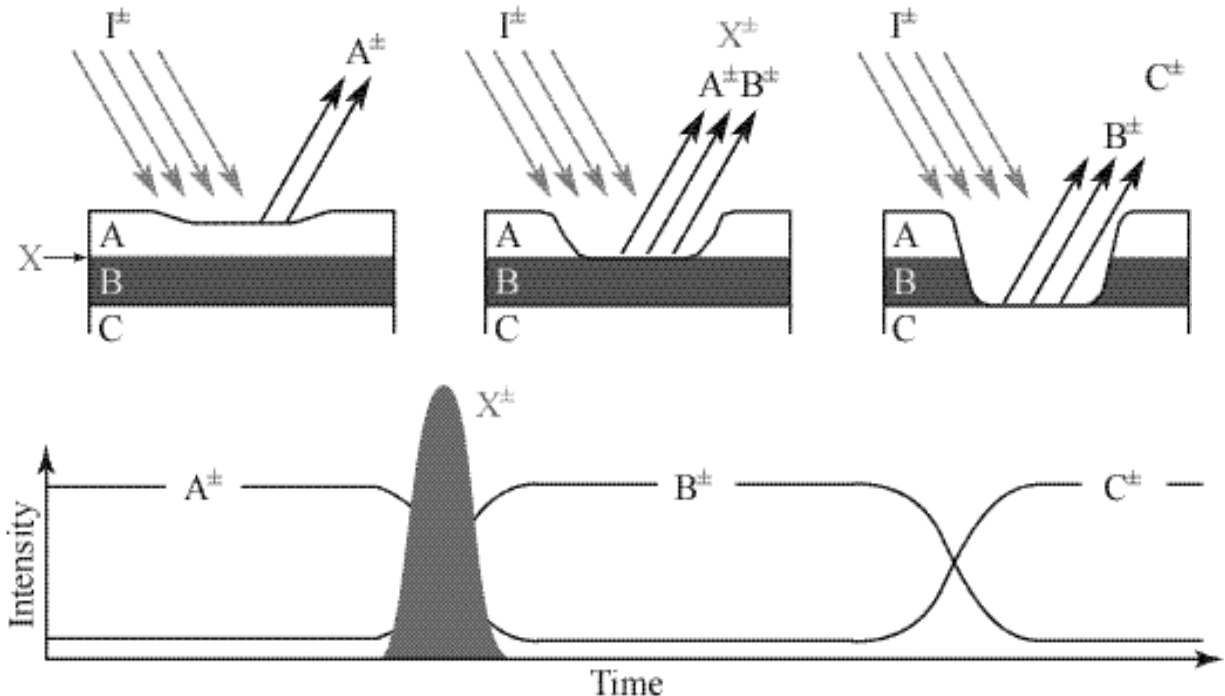


Figure 2: SIMS depth profiles are formed by collecting secondary ion intensity versus time information as the primary ion beam sputters through a multi-layer structure.

continues to erode the sample surface. An intensity versus time depth profile is the result. Layers and contaminants at interfaces can be seen (Figure 2).

Dynamic and Static SIMS

The terms “static” and “dynamic” SIMS are relatively well known. The names suggest that these are two different SIMS techniques. They are not. It really refers to the damage that is done to the sample surface. If the primary ion beam is very low current, then only a fraction of the atoms and molecules on the sample surface will be disturbed. The “static SIMS limit” is defined as less than 1 in 100 surface particles are disturbed by the primary ion beam. Below the static SIMS limit, the elements and molecules that are sputter etched are representative of the elements and molecules that are on the sample surface. Static SIMS is an effective tool for the examination of surface contamination.

Dynamic SIMS occurs when the primary beam disturbs greater than 1 in 100 of the surface particles. Usually the goal is to sputter etch the surface causing erosion with the result being a depth profile. Sputter etching is a very violent process on an atomic scale. Surface elements are mixed into the substrate. Molecules are broken apart. Mixing of the substrate material can cause new molecules to form. This breaking of existing molecules into their component parts and the formation of new molecules from atomic species

makes analysis of existing molecules on surfaces and in thin films impossible. It cannot be known if the molecule was pre-existing or was manufactured by the sputtering process.

Dynamic SIMS is a highly effective depth-profiling tool. Static SIMS is very effective in the evaluation of surface contaminants and molecules. A full examination of the capabilities of both techniques is beyond the scope of the article. The rest of this discussion will focus on dynamic SIMS and the profiling of ultra-thin films.

Dynamic Sims

Instrumentation

There are two main types of instrumentation in common use today. These are called “magnetic sector” and “quadrupole”. Both of these names refer to the method of mass separation that occurs in the secondary ion beam (see Figure 1).

As the name suggests, the magnetic sector instrument uses a magnet to separate secondary ions based on their mass-to-charge ratio. The magnetic sector instrument is capable of high mass resolution that allows for the separation of elements from molecules that occur at the same nominal mass. Elemental identification is therefore less ambiguous. Good mass separation requires secondary ions with relatively high velocity, which in the case of magnetic sector instruments, is achieved with high extraction fields over the

sample surface. High extraction fields make it difficult to bombard the sample surface with low energy primary ions, important for depth profiling thin layers.

Quadrupole SIMS uses a radio frequency field to filter out unwanted secondary ions such that molecules of only one mass can reach the detector. The radio frequency field can be changed in amplitude very quickly in order to monitor another mass. Rapid mass switching makes it possible to monitor many masses together in one profile while maximizing the number of data points that are collected. The radio frequency filter is more effective if secondary ions spend more time in the field. Therefore, low velocity ions are preferred which requires low voltage extraction fields over the sample. Low voltage secondary ion extraction fields also make it much easier to bombard the surface with a low energy primary ion beam. The primary ion beam must progress through the secondary ion extraction field to reach the sample surface. A low energy field will cause less deviation of the primary ion beam making it possible to sputter the sample surface at the desired angle of incidence.

Quantification

SIMS ion yields are dependant on the element of interest, and on the matrix material from which the element is sputtered. Ion yields for various element/matrix combinations can vary by orders of magnitude, making quantification extremely

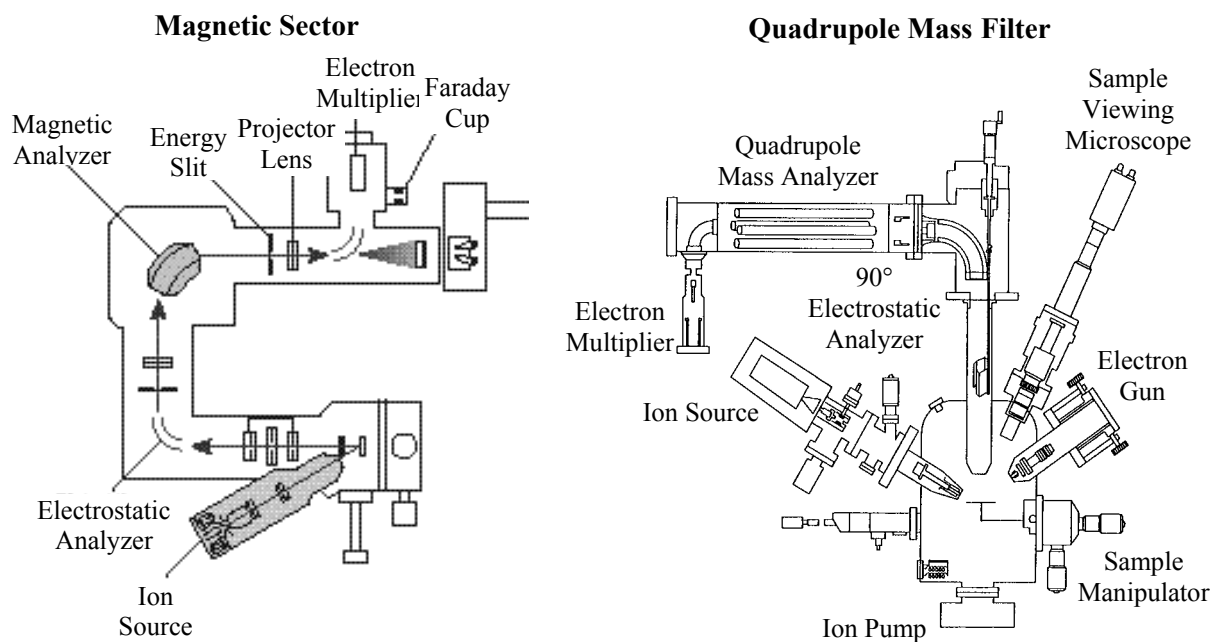


Figure 3: Diagrams of magnetic sector and quadrupole SIMS instruments in common use for dynamic SIMS depth profiling.

difficult. However, SIMS claims accuracy of better than 15% for most materials systems and better than 5% for some very specific applications. This accuracy is achieved by closely matching standards to the element/matrix material combination being examined.

The conversion of time to depth can also be done with a calibration standard, or the SIMS craters can be directly measured for depth.

Accuracy

SIMS quantification accuracy is dependant on the standard. There are a few standards for SIMS available from NIST and for these elements, accuracy is considered to be within 5% or better of the real value. Most other elements use ion-implanted standards that match the element/matrix combination that is being measured. Depth is most often determined by measuring craters with a stylus profilometer. The profilometer is calibrated using NIST traceable reference standards. Depth accuracy determined by a well-calibrated stylus profilometry is within 2%.

Precision

Reproducibility of the measurement is often more important than the absolute value of concentrations. Studies of "good vs. bad" and the effect of subtle variations in process parameters can only be seen if the measurements are very stable and repeatable. Precision is very much a product of instrument stability but careful sample placement and judicious selection of analysis location can also play a role. Typical precision using careful analysis is $\pm 5\%$. Repeat measurements on the same sample to generate some statistics can provide precision of $\pm 2\%$ or better.

References

A good practical handbook explaining most of the important considerations for SIMS analysis in more detail can be found in Reference [1]. The latest in SIMS research and development can be found in the bi-annual SIMS conferences [2].

Ultra-Thin Film Analysis

Next generation IC devices have small lateral dimensions and very thin films. Auger analysis can provide measurements in small lateral dimensions and SIMS can provide measurements in very thin layers. With recent improvements to

SIMS depth resolution, measurements on layers as thin as 1.5 nm are being made.

SIMS Depth Resolution

When an ion beam sputter etches a surface, damage is caused not only to the surface, but also to some distance under the surface. The primary beam is implanted into the sample, and a collision cascade ensues that disrupts the original positions of the atoms. It is the depth of this damage that is a prime determinant of depth resolution. To reduce the depth of this damage, the primary beam energy can be reduced so that the primary beam does not penetrate as deeply, and the ensuing collision cascade is not as energetic and does less damage. A cross-sectional diagram of this behavior is shown in Fig. 4.

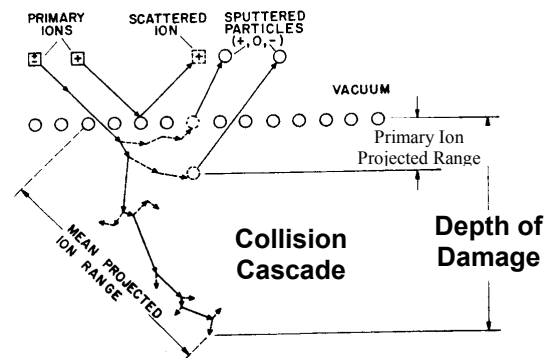


Figure 4: Damage done underneath the surface by a SIMS primary ion beam. Reduction of primary beam energy reduces this damage and improves depth resolution.

Low primary beam energy can improve depth resolution but the original surface must also be flat. Any sample surface irregularities or roughness becomes superimposed onto the crater bottom and will degrade depth resolution. If a layer of interest is particularly thin, it must be close to the surface in order to get the best depth resolution. The SIMS sputtered surface will roughen under the influence of the primary beam and depth resolution will degrade as depth increases.

Applications

The diagnostic capabilities of SIMS are generally used to evaluate the performance of fabrication tools used to make devices. That is not to say that SIMS cannot be valuable in evaluating materials performance failures. Examples will be shown.

Oxynitride

Oxynitrides or SiON is now seeing wide use as a gate dielectric material in CMOS devices. It has moderately higher capacitance than SiO₂ alone, and reduces interfacial-trapped charge. SiON thickness ranges from 5 nm down to 1.2 nm. SIMS profiles are done to measure nitrogen dose, and distribution, and to get a measure of oxide thickness. Very low primary beam energy must be used in order to get sufficient depth resolution to measure the nitrogen distribution. Quantification is done with standards that are closely matched to the concentrations and distributions of the samples being analyzed. Oxide thickness is calibrated using reference samples that are closely matched to the thickness of the measured samples. A profile from a relatively thick oxynitride film is shown in Figure 5.

Oxynitride analysis is often done to measure differences in nitrogen distribution and concentration caused by adjustments to various process parameters. High precision is therefore important in order to see the effects of these changes.

Even with very low primary beam energy, the SIMS profile still does not fully resolve the nitrogen depth distribution. For some of these oxides the total thickness consists of 5 or 6 atomic layers! The value of the SIMS profile is the ability

to see the effect on distribution and concentration of small process changes.

Ultra-Shallow Implants

Another major driver for improvement of SIMS depth resolution has been the need to characterize ultra-shallow implants, particularly boron. Much has been written and published on this subject and a full discussion is beyond the scope of this article [3]. By using an appropriate beam, energy and angle of impact, accurate information can be obtained from very shallow implants with very high depth resolution.

Ultra shallow implants are characterized for various reasons. One of the most common applications is implanter evaluation. Many shallow implants are performed by ion implanters operating in “decel mode”. Ions are extracted from a source at relatively high energy in order to get good beam current, and then decelerated to get a low energy implant. Implant systems are not able to decelerate the entire primary beam leaving a percentage that is implanted at higher energy. The SIMS depth profile is done to characterize the implant for “energy contamination”, or the amount of implant that is not implanted at the intended energy.

A profile showing energy contamination is shown in Figure 6. Data is shown on a semi-log scale, which is typical for SIMS data presentation. The

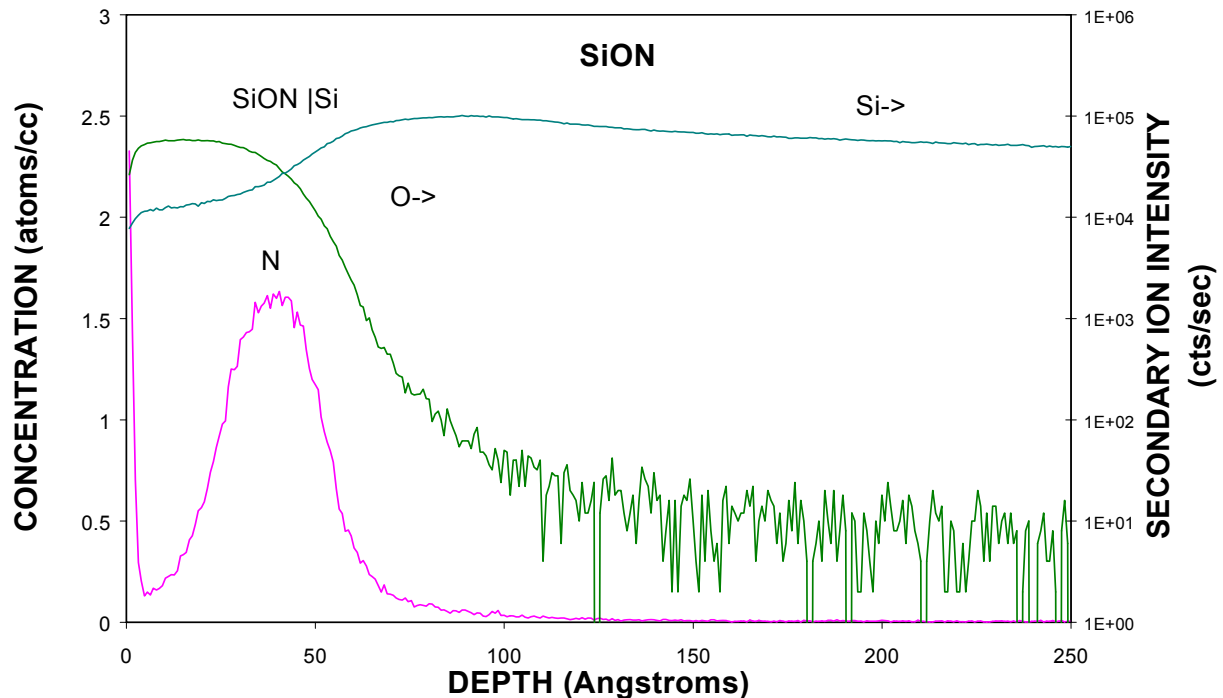


Figure 5: High depth resolution oxynitride profile showing nitrogen concentration and the position of most of the nitrogen at the oxide/substrate interface.

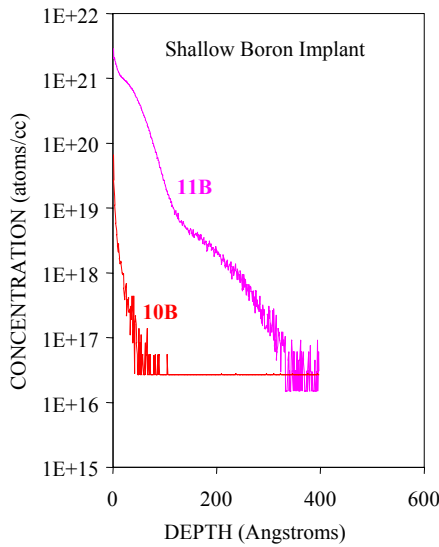


Figure 6: A boron ion implant showing energy contamination from a “decel” implanter. The curved boron distribution starting at 120 Angstroms and extending to 350 Angstroms is an energy contamination signature.

energy contamination is a dominant visual feature in the profile, but this aberration is due to the use of the log scale concentration axis. The energy contamination portion of the implanted dose is really only a few percent of the total.

Another common application is the evaluation of activation anneals. In order to make the ion implanted dopant electrically active, the sample needs to be annealed. Unfortunately, there are rapid thermal diffusion effects that make it extremely difficult to keep the implanted dopant at the implanted depth, necessary to get ultra-shallow junctions. One idea that has shown promise is laser annealing. Figure 7 shows an overlay of the original “as-implanted” profile, and the profile after laser anneal. The overlay format of data presentation allow for direct comparison of changes.

SiGe

Silicon germanium is seeing increased use in high speed, high power transistors. SiGe devices can be used instead of GaAs. Switching speed is still slower, but silicon process technology can be used to build the devices and they are easily integrated into silicon chips. SIMS plays a vital role in the development and diagnostics of deposition processes. SIMS profiles can show SiGe stoichiometry, dopant distribution, and contaminant incorporation. Thermal treatments can cause dopant diffusion and contamination can occur during epitaxial growth.

Figure 8 shows a SiGe HBT structure. High depth resolution is necessary to resolve the thin layer structure. A sharp SiGe gradient is seen which is important for device speed. Dopants in the cap

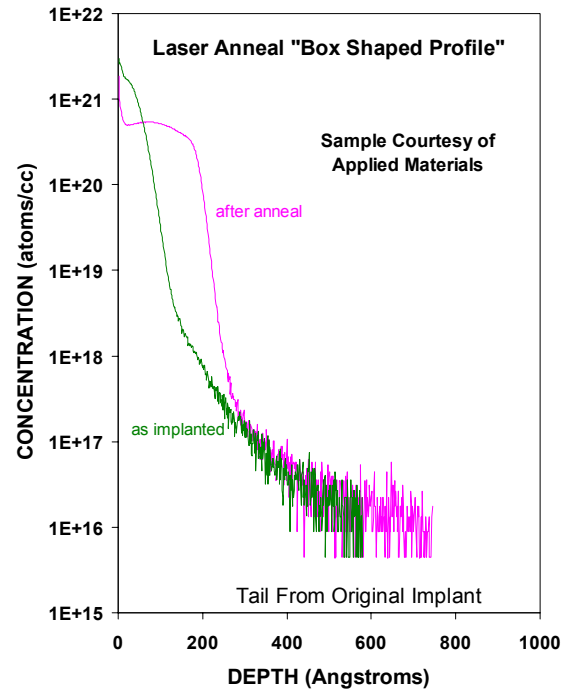


Figure 7: A shallow boron implant shown as implanted and after laser annealing. The tail is from the original implant and not from the annealing process.

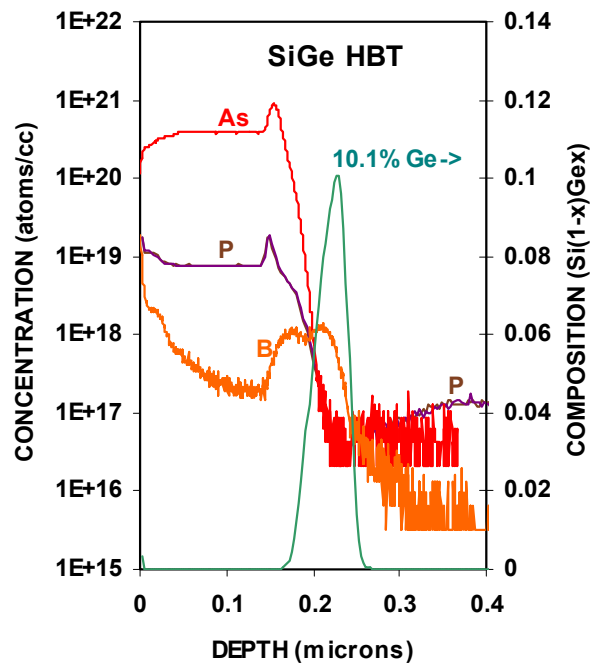


Figure 8: A high depth resolution profile showing dopant distribution and stoichiometry determination in SiGe.

layer are seen and it is important to determine their concentration, and to determine if they have diffused into the SiGe layer. Oxygen contamination can be measured at the same time, which must not exceed $1\text{E}+18$ at/cm³ or device speed is reduced.

Good information about the sample layer structure can be obtained in one profile. However, better profiles in terms of depth resolution or detection limits can be obtained using individual profiles with set-ups optimized for particular dopants or particular contaminants. For instance, the oxygen contaminant and boron dopant can be measured along with SiGe stoichiometry in one profile using a cesium primary beam. However, the depth resolution for boron will be compromised by a strong cesium primary ion beam mixing effect. Much better depth resolution for boron can be attained using an oxygen primary ion beam, but then oxygen contamination cannot be measured at the same time. A choice needs to be made about what parts of the analysis are most important.

III-V Materials

High-speed communications are highly reliant on III-V materials such as GaAs. Depth profiling using SIMS is an ideal way to characterize these multi-layer materials that often have atomically sharp interfaces. Many of the same issues that are important for SiGe are also important for III-Vs. Carbon and oxygen can act as dopants and if present in unintended concentrations, can degrade device performance. Intentional dopants need to be present in desired concentrations and

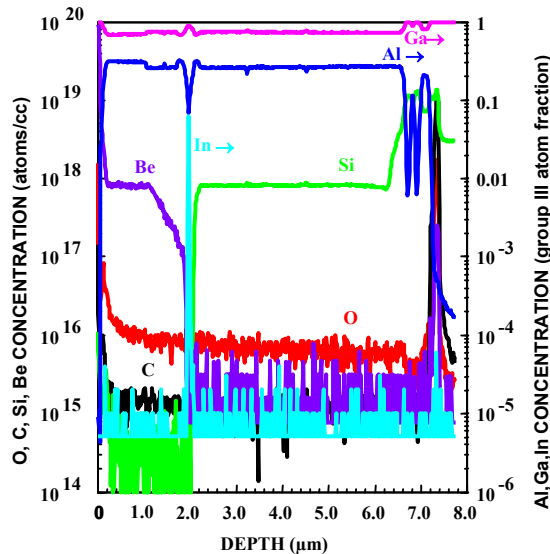


Figure 9: A depth profile of a laser structure showing the layer composition, layer thickness, dopant distributions, dopant concentrations, contaminant concentrations, and contaminant locations.

distributions. Sometimes intentional dopants will diffuse into other unintended locations. The SIMS depth profile can characterize these dopant distributions. Composition of the III-V materials can be measured, provided appropriate standards are available. III-V devices often have very complex layer structures. These can be characterized and the layer structure resolved with a depth profile. A depth profile of a III-V material can be seen in Figure 9.

Much of the SIMS work on III-V materials is as a diagnostic tool for evaluating the performance of layer growth processes and equipment, but sometimes in support of device failure analyses. Figure 10 shows an overlay of profiles taken from devices that exhibited “good” and “bad” performance. In this case a clear difference could be seen in the zinc dopant distribution.

Dielectric Materials

Dielectric materials can be evaluated with SIMS depth profiling. Electrically insulating materials can be difficult to analyze, but by using a combination of low energy secondary ion extraction fields and charge compensation provided by an electron gun, stable and reproducible depth profiling is possible. Passivation layers can be evaluated using SIMS depth profiling. In Figure 11, the layer structure of the passivation stack, aluminum contamination associated with photoresist residuals, boron and phosphorus associated with a BPSG layer, and the presence of mobile ion contaminants can all be seen. The efficacy of the BPSG mobile ion gettinger layer

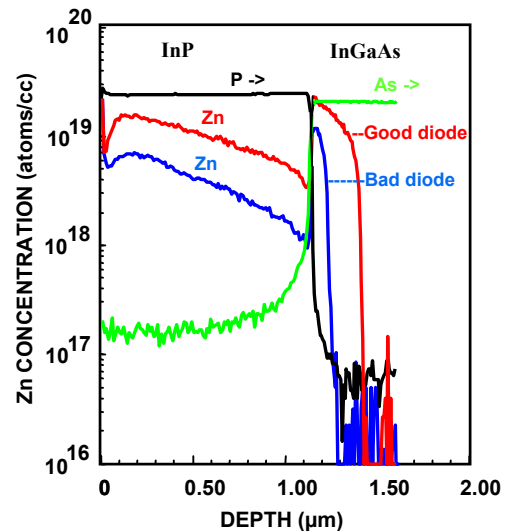
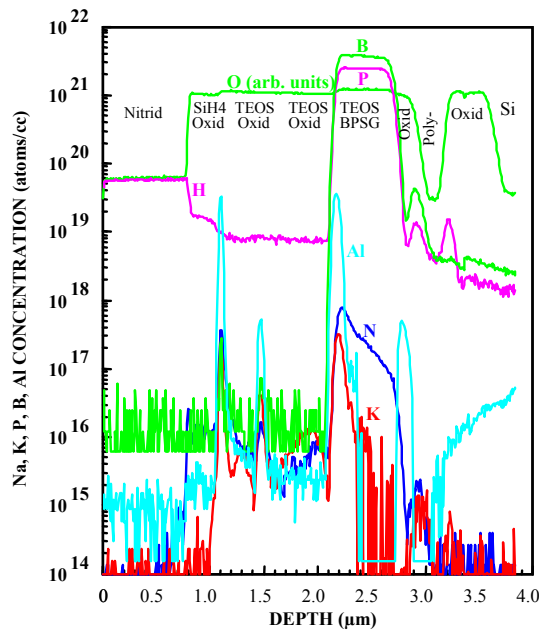


Figure 10: Overlay profiles from “good” and “bad” diodes showing large differences in the zinc dopant distribution.



can be evaluated. This measurement can be used to evaluate process failure, but more frequently is used as a quality control measurement. The quality control measurement can provide proof that the gate oxide next to the substrate is free from mobile ion contamination.

Mobile ion analysis is particularly difficult because as the name implies, elements such as Li, Na and K are mobile in SiO_2 under the influence of an electrical field. Charge compensation conditions that minimize the transport of mobile ions in SiO_2 are necessary before we can draw any conclusions about the meaning of mobile ion distributions. In the case of Figure 11, no mobile ion contamination was seen in the gate oxide providing a measure of confidence that the BPSG layer was acting as an effective getter for these elements.

Gate Oxide Breakdown

SIMS can be useful for device failure analysis. Figure 12 shows a “good vs. bad” depth profile study of gate oxides. The failed device clearly showed evidence of copper within the gate oxide that lead to breakdown, while the good device showed no evidence of copper. In this case the experiments produced a successful outcome, but the experiment must be well designed in order to be useful.

Failure Analysis Experimental Design

Dynamic SIMS for failure analysis studies is rarely

successful without some clues in advance as to what to investigate. For example, the search for an unknown contaminant at an interface will probably fail without a fairly short list of elements to investigate. The low chance of success is due to the nature of magnetic sector and quadrupole instruments. During a depth profile, elements are monitored sequentially, not simultaneously. The chances of sampling the specific element causing the problem are very low while profiling through thin films and interfaces, particularly if a long list of elements were being monitored.

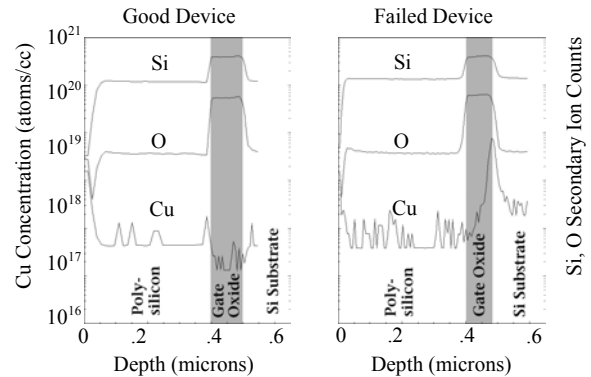


Figure 12: Depth profile failure analysis study revealing copper in the gate oxide of a failed device compared with a good device

A much more successful strategy for contaminant identification involves picking a relatively short list of elements to monitor during the profile. That will result in good sampling density for all of the monitored elements through thin films and interfaces. The list of chosen elements should include some matrix markers so that the layer structure can be identified, and a small number of suspect contaminants. The list of elements needs to be chosen by manufacturing process experts working closely with the SIMS analyst. With intimate knowledge of the process, a theory of failure can be developed, and a list of elements can be identified as most likely candidates. Working alone, it is almost impossible for the SIMS analyst to pick an appropriate list of elements. SIMS depth profiling is then done to confirm or refute various failure theories.

“Good vs. bad” experiments are an essential part of a failure analysis study. The SIMS technique is very sensitive and low levels of contaminants are found in many device structures. The fact that a contaminant was found in a layer or at an interface does not prove that it is causing the failure. Only when compared with a “good” can it be concluded that a root cause has been identified. Failure might also be due to differences in doping levels or layer structures. Again, only a “good vs. bad”

comparison study will positively identify these differences.

Failure analysis profiles often result in a null result, e.g., suspect contaminant, dopant or layer structure differences are not found when comparing “good vs. bad”. The null result holds value, however, since a theory of failure can be disproved and another theory can be investigated.

Surface and Interface Contamination

Many manufacturing processes have specifications that specify allowable levels of contaminants that may be introduced by a process. Contaminants are often introduced at the beginning or end of a process step, or during product transfer in and out of the processing vessel.

The high sensitivity of SIMS can be valuable for investigating contaminants, but there are difficulties in accurate quantification on surfaces. At the initiation of the SIMS analysis procedure, ion yields are low until the implantation of the primary ion beam element into the surface reaches high enough concentrations. During the time between the start of profiling and the maximization of secondary ion yields, called the “transient region”, quantification is difficult.

One strategy for minimizing quantification difficulties during the equilibration transient is to use an oxygen primary beam and backfill the analysis chamber with oxygen. Oxide-like ion yields are then provided from the beginning of profiling on a surface, and accurate measures of surface contamination are possible. This analysis technique is called “SurfaceSIMS” and has proven to be valuable for the study of surface contaminants, particularly from ion implanters. Figure 13 shows a SurfaceSIMS profile of aluminum contamination that was introduced onto a silicon surface during the implantation of phosphorous. The distribution shows that the implanter added aluminum, not just to the surface, but energetically deeper into the sample which indicated that the implanter beam contained not only phosphorous, but also aluminum. By noting the peak of the aluminum distribution, the energy of the implantation can be determined and the point in the beam line where the aluminum contamination is introduced can be deduced.

Quantification at interfaces can also be difficult. The matrix material plays a strong role in the ionization probability of sputtered particles. If the matrix material changes from one layer to the next, then ion yields will change, sometimes by orders of magnitude from one layer to the next.

Quantification of contaminants at the interface between layers of different materials can therefore be very complicated.

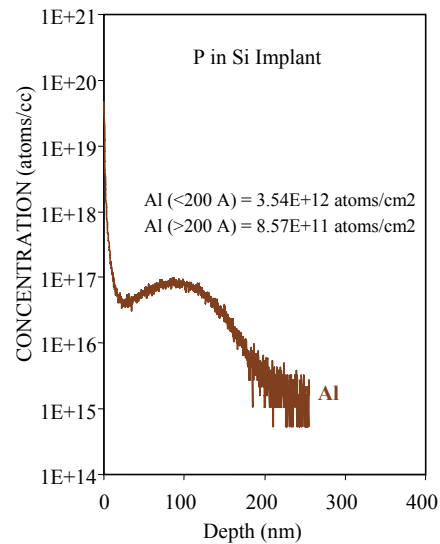


Figure 13: Aluminum contamination deposited on the surface and implanted energetically during a phosphorous implantation process.

One way to check, especially for start-of-process and end-of-process contaminants, is to deposit several layers of material using the same process step being investigated. Using this technique, interfaces will be between layers of the same material and quantification will be straightforward, provided a standard is available. Figure 14 shows multiple layers of tantalum deposited by repeatedly

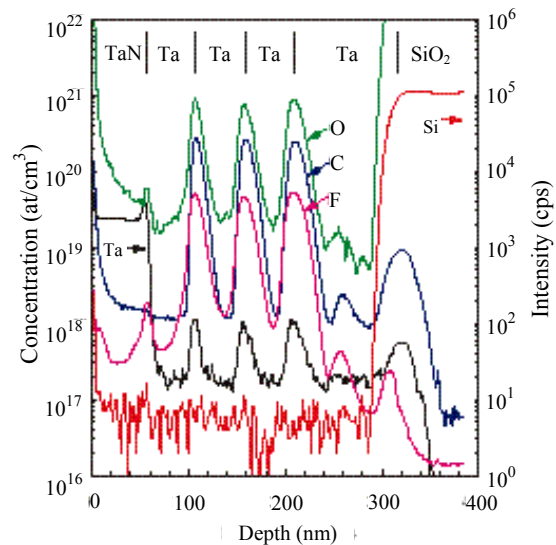


Figure 14: Carbon, oxygen and fluorine contamination are seen at interfaces between layers generated by cycling the sample through the same process. Most contamination is introduced at the start and end of the deposition process.

using the same process steps. Contamination associated with the start and stop of the process can be seen accumulated at the tantalum layer interfaces. Contamination during deposition appears to be relatively low. Because each interface is between the same materials, no large changes in ion yields are seen and the quantification of the contamination will be accurate.

Conclusion

SIMS is a powerful tool for the investigation of dopant distribution, contamination, and layer structures, and can be an effective tool for failure analysis, especially when performing “good vs. bad” studies where high sensitivity and depth profiling capabilities are required.

Successful outcomes to SIMS investigations are much more likely with careful planning before the analysis.

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