

SIMS Solutions in Materials and Life Sciences Room Great Lakes C - Session SS-TuA3

Microelectronics

Moderators: Temel Buyuklimanli, EAG Laboratories, Jang Jung Lee, TSMC

2:00pm **SS-TuA3-1 New Horizons for SIMS in the CMOS industry, Paul van der Heide, V. Spampinato, A. Franquet, IMEC, Belgium**

The Complementary Metal Oxide Semiconductor (CMOS) industry was one of the two areas that drove the development of Secondary Ion Mass Spectrometry (SIMS) in its early years, the other being geochemistry. This arose from the unparalleled sensitivity and detection limits provided by SIMS which, in the case of the CMOS industry, introduced the possibility of deriving dopant depth distributions, albeit over spatially homogeneous regions that extended to several hundred microns. Developments in SIMS instrumentation are continuing to this day, with examples ranging from the introduction of Orbitrap™ mass analyzers in lab-based SIMS platforms to the development of inline (fab-based) SIMS platforms. The former affords a ~20x improvement in mass resolution, while the later improves time-to-data. This talk will cover some examples of how these developments are opening up new opportunities for SIMS within the CMOS industry.

2:20pm **SS-TuA3-3 Self-Focusing SIMS to Enable Boron Quantification in Small Silicon Fins, Valentina Spampinato, R. Morris, W. Vandervorst, P. van der Heide, A. Franquet, IMEC, Belgium**

The continued downscaling of semiconductor devices has highlighted the importance for new metrologies to enable process control in the confined volumes and small (below 100 nm) features utilized today. Standard approaches using X-Ray Photoelectron Spectroscopy, conventional Secondary Ion Mass Spectrometry (SIMS) and Rutherford Backscattering Spectrometry lack the spatial resolution required. Alternative techniques that possess the appropriate spatial resolution e.g., Atom Probe Tomography and Transmission Electron Microscopy, are, on the other hand, time consuming and have an inherent lack of sensitivity due to the analysis volume.

In this study, the Self-Focusing (SF) SIMS concept has been successfully applied to the Boron quantification of a pattern sample composed by Silicon fins (with width size ranging from 20 to 500 nm) surrounded by SiO₂.

The spatial resolution limitation of conventional SIMS is overcome without sacrificing the excellent sensitivity by using specific cluster ions that can only originate from the Silicon fins region.

Careful study of the reference standards, such as B-doped Si and B-doped SiO₂ standards, was initially carried out in order to identify the most suitable cluster ions to use for the quantification of the Boron level. The most appropriate cluster ions were found to be BSi₂⁺ for the Boron and Si₃⁺ for the matrix.

In-situ AFM was used before and after sputtering the pattern sample, to precisely extract the sputter rate of both Si and SiO₂ regions and correctly convert the sputter time scale into depth scale.

With this approach, a Boron implant with peak concentration of ~8e²⁰ at/cm³ (average value over the different fin widths) was found, and no specific correlation with fins' size was observed. Moreover, the SF-SIMS approach was demonstrated to be in good agreement with standard SIMS approaches performed on the largest fins size, such as (1) Boron quantification after SiO₂ removal by chemical etching and (2) Boron quantification only on the fins by high lateral resolution data acquisition.

To benchmark our SF-SIMS approach, SIMS quantification was also performed on the SiO₂ region surrounding the 500 nm-wide fins and the Boron peak concentration (1.1e²¹ at/cm³) was found to be in good agreement with SRIM simulation.

2:40pm **SS-TuA3-5 Can SIMS Still Be a Relevant and Accurate Technique for Dopant Quantification and Bulk Composition of Latest Advanced Nanoelectronic Devices?, Alexis Franquet, V. Spampinato, W. Vandervorst, P. van der Heide, IMEC, Belgium**

Next generation semiconductor devices with improved performances have forced the industry to investigate and implement new materials and new devices architectures¹. Among the different materials that have attracted interest over the past years are strained-Ge and SiGe as these are good candidates for p-FET and n-FET (Field-effect transistor) thanks to their excellent hole and electron mobilities¹. The continuous downscaling of devices and the trend toward 3D architectures, lead to the deposition,

growth and integration of the different materials in more and more confined volumes (of dimensions <10nm). Therefore, characterization methods are needed that can not only provide chemical information (for bulk composition) and high sensitivity (for dopant concentration), but also do so at a spatial resolution compatible with the devices under investigation. SIMS is a well-known surface analysis technique which enables to measure the distribution of elements and molecules in 1D (depth profiling), 2D (spatial imaging) and 3D (volumetric imaging)². Since decades, SIMS was used in the Complementary Metal Oxide Semiconductor (CMOS) industry to derive dopant depth distributions thanks to its exceptional sensitivity and very low detection limits. This was until recently mostly done on blanket samples which turns to be irrelevant nowadays, as the properties of nano-volumetric devices are far away from the one of blanket samples. Although SIMS lacks the spatial resolution to directly probe devices from sub-10nm technologies, it can analyze the composition of narrow trenches (<20nm) using the concept of Self Focusing SIMS (SF-SIMS)³.

This talk will discuss both the accuracy of Boron and Phosphorous dopant concentrations/profiles and the Ge quantification in blanket and patterned SiGe structures of dimensions far below the lateral resolution of SIMS. It will be shown how the use of concepts such as SF-SIMS and the introduction of new developments in the SIMS technique such as the Orbitrap™ mass analyzer allow to extend the application of SIMS in the semiconductor industry for the next decades. Several examples will be discussed, among which the quantification of the B dopant level and Ge content in complex devices made of SiGe-B epi dots (Ø<50nm) grown on 10nm wide Si:B nanowires.

¹ R. Chau, Process and Packaging Innovations for Moore's Law Continuation and Beyond, IEEE IEDM Tech. Dig. (2019) 1.1.1

² P.A.W. van der Heide, Secondary Ion Mass Spectrometry: An Introduction to Principles and Practices, John Wiley & Sons (2014) ISBN 978-1-118-48048-9

³ A. Franquet, W. Vandervorst et al., Appl Surf Sci 365 (2016) 143-152

3:00pm **SS-TuA3-7 Characterization of GaN HEMT Structures by Combined SIMS & HAXPES Approach, Tarek Spelta, M. Veillerot, E. Martinez, P. Fernandes Paes Pinto Rocha, L. Vauche, CEA/LETI-University Grenoble Alpes, France; B. Salem, CNRS-LTM, Université Grenoble Alpes, France; B. Hyat, CEA/LETI-University Grenoble Alpes, France**

III-N materials are gaining interest because they are widely used in high-tech industry. For instance, High Electron Mobility Transistors (HEMTs) with AlGaN/GaN structure are under development for devices in power electronics. The presence of a twodimensional electron gas (2-DEG) at the AlGaN/GaN interface allows greater electron mobility, which makes them very interesting for applications. The final properties of a device strongly depend on the quality of the interface between the GaN and the dielectric.

The issues to understand on this interface are the presence of deleterious Gallium oxide that lead to current leaks and the presence of undesired contaminations such as C, Cl, H, related to the manufacturing process. It is therefore necessary to characterize this interface with an analysis technique that illustrates with extreme precision the elements composition. Secondary Ion Mass Spectrometry (SIMS) appears to be particularly adapted to investigate such complex interfaces. In particular, low energy Caesium sputtering appeared to be an adequate path to depth profiling of III-N structures¹.

This research work illustrates the SIMS depth profiling of Al₂O₃/GaN stacks, where a 10 nm thick Al₂O₃ layer was deposited by ALD on top of as-epi and etched GaN surfaces. The use of two oxidant precursors such as O₃ and H₂O was investigated. The presence of Gallium oxide at the interface was investigated in the light of what was obtained for a Al₂O₃/GaO_x/GaN stack where a 4 nm-thick GaO_x layer was grown prior to alumina deposition.

Dual beam depth profiling is performed with a TOF-SIMS 5 (from IONTOF GmbH) using monoatomic Caesium sputtering beam at 500 eV, whereas the analysis is carried out using Bi₃⁺ at 15 KeV. Depth profiling is also investigated through a Magnetic SIMS on SC-Ultra instrument (from CAMECA), with monoatomic Caesium beam at 250-500 eV used for both sputtering and analysis. Benefits and peculiarities of both the techniques on III-N structures are discussed. The results highlight the different Al₂O₃ structures impact oxidation and the presence of impurities at the interface.

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Furthermore, these buried interfaces were explored down to 25 nm through hard X-ray photoelectron spectroscopy (HAXPES), and information about their chemical composition were provided. Finally, AFM roughness measurements before and after SIMS analysis were conducted to have morphologies information on samples.

Acknowledgments:

This work, carried out on the Nanocharacterization Platform of Minatec (PFNC), was supported by the "Recherche Technologique de Base" program.

Reference:

1. Kachan, M., et al. "O²⁺ versus Cs⁺ for[...]devices. " *Applied surface science* 231 (2004)

3:20pm SS-TuA3-9 The Implementation of ToF-SIMS in the Development of State of the Art Ohmic Contacts to GaN, Tatyana Kravchuk, Technion, Israel; *Z. Fogarassy*, Institute for Technical Physics and Material Science, Centre for Energy Research, Budapest, Hungary; *A. Rácz*, Institute for Technology Physics and Material Science, Centre for Energy Research, Budapest, Hungary; *A. Wójcicka, M. Borysiewicz*, Institute of Microelectronics and Photonics, Warsaw, Poland; *S. Grzanka*, Top-GaN Ltd., Warsaw, Poland; *P. Perlin*, Institute of High Pressure Physics PAS, Warsaw, Poland

For the performance and reliability of semiconductor devices stable contacts at interfaces (with low contact resistance and linear I-V behavior) are crucial. Their preparation and characterization are dominant issues in the microelectronic industry. Ohmic contacts are usually created by depositing thin metal films with a specifically chosen composition followed by annealing. The contact resistance strongly depends on the interface quality and chemical state which makes the design and fabrication of low resistance contact structures such a challenge for microelectronics.

One of the best techniques to investigate the material processes occurring in contact structures is TOF-SIMS. The technique is able to detect all elements (even the light ones) with a high mass resolution, mass accuracy, and good signal-to-noise ratio. In a dynamic mode, TOF-SIMS has a monolayer resolution and micron depth range which makes it indispensable for the investigation of diffusion and layer intermixing, often arising during contact formation and aging.

Transparent or semi-transparent contacts are necessary for many applications like laser diodes or photovoltaics. The most popular choice is ITO, however alternative material systems gain interest because of sustainability and difficulties with indium sources. In this work, we investigated one of such systems which is based on zinc oxide (ZnO), which becomes conductive after doping with Al. In addition, if ZnO is in a solid solution with Mg the bandgap will widen. To investigate the interface between the ZnMgO:Al and the GaN substrate we checked a number of subcontact layers and annealing temperatures while using I-V measurements as a reference for the quality of the contact. By comparing the ToF-SIMS measurements of ohmic and non-ohmic structures we suggested the mechanism explaining the processes of contact creation. The results are supported by TEM and XRD results.

4:00pm SS-TuA3-13 Transfer of Zirconium Oxide Nanotubes onto Zirconia-Based Ceramic Implants, Swathi Naidu Vakamalla Raghunath, University of Siegen, Germany

Nanostructured architectures, offer the possibility of creating storage units whilst improving bio-integration and functionality as a result of superior adhesion and robust reactivity due to the increased surface area.[1,2] Electrochemical anodization is an efficient way to develop large-scale nanostructures on a material's surface, unfortunately fabrication by anodization is restricted to valve-metals. Non-metals, especially biomaterials are often metal-oxides and ceramics such as in the field of dental and orthopedic applications.[3] In order to develop nanoporous or nanotubular surface structures on such surfaces, multi-step procedures can be applied, starting with metal deposition on the parent materials via vacuum assisted treatments such as ALD, e-beam sputtering, FDM, etc.[4-6] This approach poses numerous challenges, such as adhesion of deposited metal to the ceramic substrate, stability at the interface in addition to the cost-factor to name few. Herein, we demonstrate the possibility of attaching ZrNTs to ZrO₂ ceramics without the use of any intermediate treatment of the parent-ceramic. We report on the synthesis

route for metal-oxide nanotubes via electro-chemical anodization of zirconium foil resulting in the formation of zirconia nanotubular (ZrNT) films, that are subsequently transferred onto pre-formed zirconia (ZrO₂) implant material. This approach involves a direct transfer of ZrNT films onto the ceramic implant via an acetone bath after a successful detachment from the foil using office-adhesive tape prior to transfer. This simple technique is not limited by geometric constraints of the parent material.

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4:20pm SS-TuA3-15 Quantification of Sims Measurements by Using Ion Implanted Metallic Standards, Guiomar D. Soria, M. González, CIEMAT, Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas, Spain; *M. Crespillo, G. Garcia*, CMAM, Centre for Micro Analysis of Materials, Spain

Secondary ion mass spectrometry (SIMS) is a powerful analytical technique for surface characterization, allowing the identification of elements and isotopic species present in the solid composition with high sensitivity. SIMS analysis is widely used for materials with applications in microelectronics, aeronautics, and fusion, among others.

Nonetheless, it is well-known that the quantification of this method is challenging. The reason is that the signal of secondary ions sputtered from the substrate by irradiation with the primary ion beam is strongly influenced by the chemical environment of the solid. I.e., the analyte of interest will be easy/difficult to sputter and ionize depending on the type of bonding with the matrix. This fact is reflected in the measurement with greater or lesser signal intensity. This phenomenon called the "matrix effect" hinders reaching a general quantification expression for the technique, making it difficult to correlate the intensity response of the registered elements with their accurate concentration in the substrate.

In this contribution, a solution to minimize the matrix effect for SIMS analysis quantification is investigated through the production of calibrated standards. For this aim, implanted ions with known concentrations in a fusion-relevant matrix are applied and correlated with the SIMS signals. Specifically, the application of this methodology is now focused on a metallic substrate, with a particular interest in the fusion field, implanted with some elements with easy ionizability, where the parameters of concentration and depth of the ion into the substrate are controlled. Therefore, standards consisting of tungsten metal commercial matrices were prepared by ion implantation with 18 MeV iron and 10 MeV chromium ions at two different fluences (about 10¹⁵ and 10¹⁶ ions/cm²) at room temperature. Subsequently, the implanted samples were characterized using surface and depth profile SIMS measurements for identifying the Fe and Cr ions recorded signal. The accurate value of the ion doses was obtained using Rutherford backscattering (RBS) analysis on control silicon substrates. The resulting calibration concentration curves were associated with the SIMS signals, being the tool for the quantification analyses of unknown concentrations of Fe and Cr in test substrates. Therefore, the method was validated by analysing impurities and alloying of these elements in a non-commercial tungsten matrix.

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4:40pm SS-TuA3-17 Ion Implantation Applications for In-Line SIMS Metrology, Lawrence Rooney, S. Okada, Nova

In the semiconductor industry, ion implantation process has expanded to a wide range of applications with doses and energies spanning several orders of magnitude.

Ion implantation is a very complicated process with many parameters and factors that affect the implant profile. For example, shadowing effects from higher aspect ratio of photoresist opening, ion channeling or de-channeling effects due to implant angle variations, and dose and implant energy accuracies are all important factors in achieving uniform device performance and good product yield. In addition, current process controls are done on test wafers with certain time intervals, where broken sample pieces are sent outside of the fab for SIMS analysis. The turnaround time is generally long, and the results often do not reflect the actual production conditions. It is known in some cases that, while the control charts are in good standing, the product has failed to meet its specification. The demand for consistent implantation material is becoming more and more important. Hence, the desire for better implant process control is sorely needed.

This paper explores how utilizing Secondary Ion Mass Spectroscopy, (SIMS) in-line to measure peak concentration, peak depth, and dose simultaneously to provide better implant process control.

5:00pm SS-TuA3-19 Molybdenum Oxide Substrate Used in "Storing Matter" SIMS Technique – Determination of Relative Sensitivity Factors of 20 Elements, Piotr Konarski, J. Ażgin, Łukasiewicz Research Network - Tele and Radio Research Institute, Poland; M. Kasik, MK2 Technologies, Inc.; H. Brongersma, Eindhoven University of Technology, Netherlands

We present the use of molybdenum oxide substrate as a collector plate in storing matter (SM) technique applied in SIMS [1, 2] and determination of relative sensitivity factors (RSF) of this technique for a series of 20 elements from Mg to Bi.

The SM technique enables quantitative SIMS analysis by separating process of sputtering and process of secondary ion formation. The analysis is done in two steps, first the sputtered material of ion bombarded surface is deposited onto the substrate so as to obtain approximately a sub-monolayer coating. Then, the substrate with the stored material is analysed using a classical SIMS analytical method.

As substrates we use 300 nm thick molybdenum oxide MoO_{3-x} layers deposited onto titanium plates by high-vacuum evaporation of ultra-pure MoO_3 . The SM experiments are carried out in Hiden SIMS Workstation apparatus equipped with a special sample manipulator enabling positioning of samples for sputter deposition process and positioning of the collector plate for SIMS analysis of the deposited material.

In SM experiments we use 5 keV, 48 nA O_2^+ beam. During sputter-deposition process this beam is scanned over $600 \times 600 \mu\text{m}$ area of the sample for a time period of 540 s, equal for each analysed sample. Then the SIMS analysis of stored material is performed, and this beam is scanned over $3000 \times 3000 \mu\text{m}$ area of the collector plate. Obtained results show, that most of the examined elements yield higher SM SIMS signals comparing to classical SIMS analysis of this set of elements.

The obtained results allow to calculate the RSF factors of the 20 elements deposited on MoO_{3-x} substrate. We plot the obtained RSF values versus atomic mass, and versus first ionization potential values of the examined elements and compare the plots with typical SIMS RSF factors of elements implanted into silicon matrix. We also compare the results with RSF values of another analytical technique - glow discharge mass spectrometry (GDMS) [3].

Authors thank The National Centre for Research and Development, Poland for funding the project PL-TW/VII/4/2020 in years 2020-2022.

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5:20pm SS-TuA3-21 CsM^+ Cluster Method in Dynamic SIMS: A Versatile and Practical Approach for Thin Film Electronic Materials, Marinus Hopstaken, S. Molis, IBM T.J. Watson Research Center

Reactive Cs^+ primary beam is routinely employed in SIMS depth profiling for reasons of negative ion yield enhancement. Alternatively, positively charged Cs -cluster ions (Cs_nM^+ ; $n = 1, 2$) can be analyzed, providing a

versatile approach for simultaneous detection of either electro-positive (CsM^+) and electro-negative species (Cs_2M^+) [1]. Normalization of Cs_nM^+ cluster ion to their Cs_n^+ reference ions is commonly applied to reduce so-called 'matrix effects' [1]. Here, we will outline practical considerations, quantification aspects, and some artifacts in Cs_nM^+ cluster analysis. We will demonstrate this using various examples of Cs_nM^+ depth profiling of thin-film nano-structured materials for a variety of electronic (CMOS, memory) and post CMOS (III-V, quantum computing) applications.

Application of Cs_nM^+ cluster approach for dopant profiling in Si is generally compromised due to poor sensitivity [2]. In contrast, we have reported excellent sensitivity for p+ dopants (Mg, Zn) in high-mobility III-V compounds. Useful yields for Mg and Zn are found to be largely insensitive to the matrix composition for different binary and ternary III-V materials (i.e. $\text{In}_x\text{Al}_y\text{Ga}_{1-x-y}\text{As}$). Quantitative analysis down to $5 \times 10^{15} \text{at.cm}^{-3}$ detection limits in III-V, using appropriate ion implant calibration standards.

Cs_nM^+ cluster approach is well suited for quantitative analysis of simple binary alloys such as $\text{Si}_{1-x}\text{Ge}_x$ and NiSi silicides [3,4]. Quantification is based on linearization of SIMS $\text{Cs}_n\text{M}^+ / \text{Cs}_n\text{Si}^+$ ion intensity ratios to the $[M]/[\text{Si}]$ atomic ratio, derived by absolute external methods such as XRD, RBS, XRR). Here we extend this approach to more complex (quasi-) ternary mixtures such as group III-arsenide or phosphide compounds (e.g. $\text{Al}_x\text{Ga}_{1-x}\text{As}$) and phase change materials (PCM). We generally find good linear correlation between CsM^+ ion intensity ratios and their corresponding atomic ratios.

We generally employ the Cs_nM^+ cluster approach to more complex multilayer structures for the simultaneous analysis of electropositive (CsM^+) and electronegative species (Cs_2M^+), while facilitating robust and reproducible charge compensation in thin dielectric layers in a magnetic sector instrument. We will demonstrate examples across a wide range of applications (high-k / metal gate, alternate materials for memory applications, etc...) to demonstrate versatility and general applicability.

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