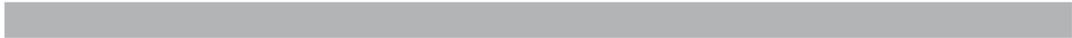


FRONT-END PROCESSING METROLOGY PROGRAM

The dimensions of the active transistor areas are approaching the spacing between dopant atoms, the stochastic regime, complicating both modeling and doping gradient measurements. Thin dielectric and conducting films are approaching monolayer thicknesses.

As device dimensions continue to shrink, junctions and critical film thicknesses approach the realm of several atoms thick, challenging gradient, thickness and wafer flatness and roughness metrology as well as electrical and reliability characteristics. The gate dielectric, traditionally SiO_2 , will soon no longer be viable. The overall task is to provide starting wafer dimensional and defect metrology, suitable metrology and reference materials for their dielectrics and junctions, including electrical characterization, gradient, thickness and roughness metrology and overall reliability metrology.



WAFER AND CHUCK FLATNESS METROLOGY

GOALS

Develop measurement support for 300 mm diameter silicon wafers used in lithography applications. This project provides measurement and technology infrastructure to support the measurement of wafer thickness variation of 300 mm silicon wafers, and surface flatness of chucked wafers.

CUSTOMER NEEDS

Decreasing linewidths and the accompanying reduction in depth of focus, larger wafer diameters for current stepper lithography applications, and the advent of immersion lithography place ever increasing restrictions on wafer flatness and the required measurement uncertainty. The International Technology Roadmap for Semiconductors (ITRS) projects a flatness of ≤ 51 nm at the die site for the 65 nm node by 2010. We are focused on meeting customer requirements for calibrated thickness variation maps of free form wafers and flatness measurements of chucked wafers. We are addressing the need for standard 300 mm wafers with calibrated thickness variation with the NIST Improved Infrared Interferometer (IR³). These independent, traceable wafer thickness calibrations enable manufacturers of wafers and wafer metrology instruments to certify the performance of their metrology instruments. In addition, thickness variation measurements of silicon wafers can be combined with models of wafer/chuck interactions to determine the flatness of low surface area wafer vacuum chucks, which is difficult to measure directly. The surface flatness of chucked wafers can be measured using NIST's "eXtremely accurate CALIBration InterferometER" (XCALIBIR). XCALIBIR has a 300 mm aperture for flat measurements and provides a way of verifying models of wafer/chuck interactions.

TECHNICAL STRATEGY

1. The IR² is an infrared phase-shifting interferometer, operating at a wavelength of 1550 nm that measures the thickness of low-doped silicon wafers up to 300 mm diameter (see Fig. 1a and Fig. 1b). The interferometer may be used in several configurations with collimated and spherical test wavefronts. The collimated wavefront mode is the current focus of the project. In this method, the planar infrared wavefront is normally incident on the wafer. A portion of the beam is re-

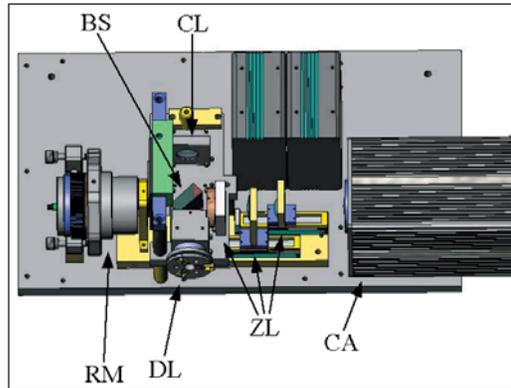


Figure 1a. NIST's infrared interferometer (IR²). The main components of the interferometer are: collimator lens (CL), polarizing beam splitter (BS), phase-shifting reference mirror mount (RM), diverger lens (DL), zoom lens system (ZL), and camera (CA).



Figure 1b. Test end of the IR² setup for TTV measurements of 300 mm wafers. The picture shows the collimator lens together with a silicon wafer and a return flat.

flected from the front wafer surface, while the rest passes through the wafer and reflects from the rear surface. The interference of these two wavefronts produces fringes and, by wavelength phase shifting, allows calculation of the wafer thickness variation. Figure 2 (pg. 66) shows the thickness variation map for a 300 mm silicon wafer.

Technical Contacts:
U. Griesmann
R. Polvani

"NIST continues to support the effort to bring quantitative standards and measurement practices to the semiconductor wafer metrology area. Their support in the area of wafer-chuck interaction studies are enabling advances in the state-of-the-art of wafer chucking that are essential to fully realize the potential of short wavelength lithography. WFSI's ability to collaborate with NIST in this area is critical to us."

T. D. Raymond
Wavefront Sciences, Inc.,
Albuquerque, NM

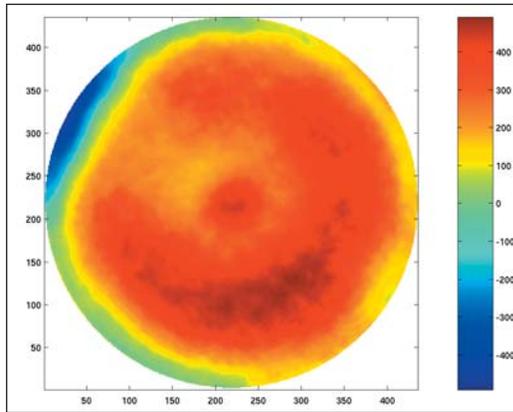


Figure 2. Thickness variation, with piston term subtracted, of a 300 mm double-side polished silicon wafer measured with IR². The height unit is nm, the coordinates in x- and y-direction are detector pixel numbers.

DELIVERABLES: Install interferometer in clean-room for calibration measurements, 4Q 2005. Develop complete uncertainty analysis for thickness and thickness variation measurements, 2006.

2. A component in the evaluation of chucked wafer non-flatness is the characterization of interactions between the vacuum chuck and wafer. We are collaborating with Wavefront Sciences, Inc. (WFSI), Albuquerque, New Mexico and potentially one or more lithographic stepper manufacturers to help understand these interactions. WFSI is carrying out numerical analyses of the chuck/wafer interface for various chuck geometries. Figure 3 shows a model of XCALIBIR, a general purpose 300 mm aperture phase measuring interferometer developed at NIST, which is used to measure the flatness of chucked wafers. The are then be used to evaluate the influence of wafer/chuck interactions on the chucked wafer flatness. (FIGURE 3)

ACCOMPLISHMENTS

- IR² has undergone a major upgrade that enables us to address the metrology needs for 300 mm diameter wafers. A collimator lens has been installed that can illuminate the entire surface of a 300 mm wafer and thus allows us to make a measurement of the wafer's thickness variation in a single measurement. The imaging system of the interferometer now can measure wafers with larger slopes and the spatial resolution of the detector was doubled. Further improvements will be aimed at reducing the noise level and at improving the measurement uncertainty.

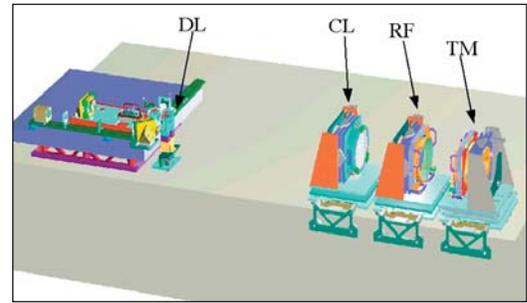


Figure 3. XCALIBIR interferometer configured for flatness measurements. The main components of the interferometer, from left to right, are: breadboard with source and imaging optics, diverger lens (DL), collimator lens (CL), reference flat (RF), and test mount (TM). For silicon wafer flatness measurements a wafer chuck is mounted on the test mount.

- The optical components in the IR² interferometer were improved to reduce the measurement noise. Wavelength phase-shifting has been implemented and a TTV map repeatability of 5 nm peak-valley has been achieved for 300 mm wafers.

- The IR² interferometer is being moved to a clean-room which will allow us to make calibration measurements of wafers supplied by industry customers.

- The flatness of 200 mm and 300 mm diameter wafers in the chucked condition was explored using the XCALIBIR interferometer.

COLLABORATIONS

1. WaveFront Sciences, Flatness measurements of free form and chucked wafers for metrology tool validation.
2. MEMC Electronic Materials, Wafer thickness standard development.
3. Siltronic, Wafer thickness standard development.
4. Intel, Development of very thin silicon thickness standards.
5. Frontier Semiconductor, Flatness metrology for patterned wafers.

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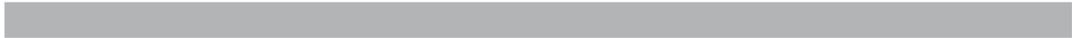
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MODELING, MEASUREMENTS, AND STANDARDS FOR WAFER SURFACE INSPECTION

GOALS

Provide industry with models, measurements, and standards for particles and other defects in order to improve the inspection of wafer surfaces. Develop facilities to accurately measure particle size and to deposit monosize particles on calibration artifacts to reduce the uncertainty in the sizes of particles used by the semiconductor industry to calibrate scanning surface inspection systems (SSIS). Investigate theoretically and experimentally the behavior of light scattering from particles, defects, and roughness on wafer surfaces.

CUSTOMER NEEDS

The Semiconductor Industry Association's (SIA) International Technology Roadmap for Semiconductors identifies the detection and characterization of defects and particles on wafers to be a potentially show-stopping barrier to device miniaturization. The roadmap specifies polystyrene latex (PSL) equivalent diameter particles that must be detectable on bare silicon, nonmetallic films, metallic films, and wafer backsides each year. Currently, no solutions to this inspection problem exist now for particles on bare silicon and on non-metallic films, while solutions do not exist for wafer backsides and metallic films in 2007 and 2009, respectively. While the detection sensitivity for defects must be increased, the ability to characterize defects in terms of size, shape, composition, etc., is critical for yield-learning. Defects must be characterized independent of defect location and topology.

With the need to detect smaller defects, the costs of inspecting wafers are skyrocketing. In order for new advances to be implemented in production environments, improvements in sensitivity must be achieved without suffering a tradeoff in throughput and must be cost-effective. The drive towards *in situ* sensors for production tools requires techniques which can be effectively miniaturized.

In order that wafer manufacturers and device manufacturers have a common basis for comparing specifications of particle contamination, improved standards for particles are needed. A recent comparison of the measurements of calibration wafers by 13 different Scanning Surface

Inspection System (SSIS) indicated unacceptably large deviation between the SSIS results and the actual particle sizes. This study involved six particle sizes ranging from 88 nm to 290 nm and included the NIST SRM 1963 and two other sizes measured by NIST. For the two smallest particle sizes, 88 nm and 100.7 nm, the scanners systematically underestimated the size by about 8 %. By 2005, it is anticipated that accurate calibration particles as small as 30 nm will be needed.

By 2010, at the 45 nm node, particles having diameters 22.5 nm must be detectable on bare silicon and nonmetallic films, 36 nm on metallic films, and 45 nm on the backsides of wafers. No known solutions exist at this time. [2004 ITRS, Yield Enhancement, Table 112b]

TECHNICAL STRATEGY

There are two major strategies for improving the performance of scanning surface inspection systems. One strategy is to develop a fundamental understanding of optical scattering at surfaces so that tool manufacturers can optimize the performance of their instrumentation, in terms of defect detection limits and discrimination capabilities, to characterize the response of instrumentation to different types of defects, and to develop and calibrate particles of well-defined size and material. Recent work by this group has demonstrated that the polarization of light scattered by particulate contaminants, subsurface defects, and microroughness has a unique signature that can be used to identify the source of scatter. In particular, it was found that small amounts of roughness do not depolarize scattered light. This finding has enabled the development of instrumentation which can collect light over most of the scattering hemisphere, while being blind to microroughness. That instrumentation, for which a patent has been awarded, should result in a factor of two improvements in minimum detectable defect size.

A second strategy is to provide leadership in the development of low uncertainty calibration particles for use in calibrating surface scanners. A major focus has been development of the differential mobility analysis (DMA) method for accurately sizing monosize polystyrene spheres. This work together with a SSIS round robin has

Technical Contacts:

T. A. Germer
G. W. Mulholland

"I would like to thank you and NIST for the support that you have provided to VLSI Standards in the sizing of polystyrene latex spheres through the work of Dr. George Mulholland. We are very pleased with the measurements that Dr. Mulholland has performed, and with the level of technical support that the NIST staff has provided to us. This work is of technical and economic importance to the semiconductor industry and to VLSI Standards, because the ability to correctly size ever smaller particulate contaminants on silicon substrates is key to the manufacturing yield of silicon chips. We look forward to a continuing technical relationship between VLSI Standards and NIST."

*Marco Tortonese, Ph.D.
VLSI Standards, Inc.*

provided evidence that current SSIS measurements have an unacceptably large uncertainty for particle sizes in the 90 nm to 100 nm size range. The technical focus of our future work will be applying the DMA for accurately sizing calibration particle sizes as small as 30 nm, developing methods for generating other types of monosize particles, and developing laser surface scattering methods for quantitative particle sizing.

Specific project elements are defined below:

1. Polarized Light Scattering Measurements

– The Goniometric Optical Scatter Instrument (GOSI) enables accurate measurements of the intensity and polarization of scattered light with a wide dynamic range, high angular accuracy, and multiple incident wavelengths (visible and UV) (see Fig. 1). We measure the light scattering properties of well-characterized samples exhibiting interfacial roughness, deposited particles, subsurface defects, dielectric layers, or patterns. The emphasis is on providing accurate data, which can be used to guide the development of light scattering instruments, and to test theoretical models.

DELIVERABLE: Measurement capability for measuring diffuse and diffracted light from a grating. 4Q 2005

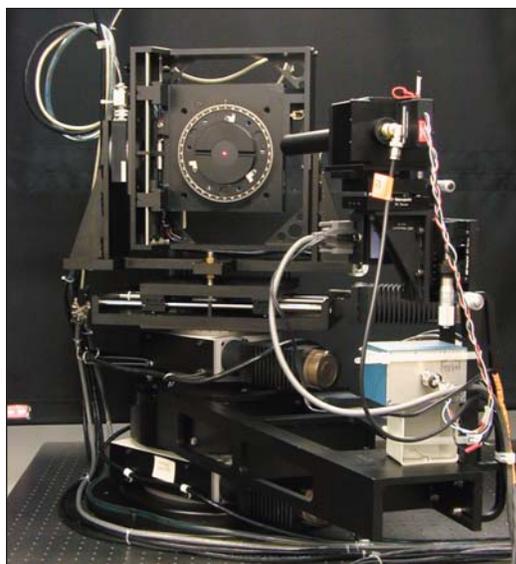


Figure 1. The Goniometric Optical Scatter Instrument is a state-of-the-art laser scattering facility.

2. Theoretical Light Scattering Calculations

– The focus of our theoretical work is on (a) developing models that accurately predict the polarization and intensity of scattered light, and (b) determining what information can be efficiently

and accurately extracted from light scattering measurements. Approximate theories are used in conjunction with more complex techniques to gain an understanding of which parameters affect the light scattering process. Particular cases that are being analyzed include: (a) scattering by defects and roughness associated with dielectric layers, (b) scattering by particulate contamination on bare and oxidized wafers, and (c) scattering by periodic structures.

DELIVERABLE: Publish computer code for scattering by axially symmetric particles on surfaces. 3Q 2005

3. Size Distribution Measurements

– Differential mobility analysis (DMA) has been shown to be capable of making accurate size measurements for mean particle size for 100 nm monosize polystyrene spheres. There are promising results for the measurement of the size distribution for broader size distributions; however, the results are not quantitative. Work is in progress to quantify the uncertainty in the size distribution measurement and to extend the method to smaller particle sizes.

DELIVERABLES: Certify a 100 nm particle size SRM to replace SRM1963 using the NIST particle sizing calibration facility. 2Q 2005

Certify a 60 nm particle size SRM using the NIST particle sizing calibration facility. 2Q 2005

4. Aerosol Generation

– An aerosol must be formed typically from a liquid spray of a particle suspension before the particles can be sized by the DMA or deposited on a wafer. Work is in progress to use a variety of innovative methods for generating, shaping the size distribution of the aerosol, and depositing the particles. These include the electrospray for generating particle sizes smaller than 60 nm, impactor to remove the large size fraction of the aerosol and to deposit the particles, and an electrostatic chamber for depositing small particle sizes (see Fig. 2).

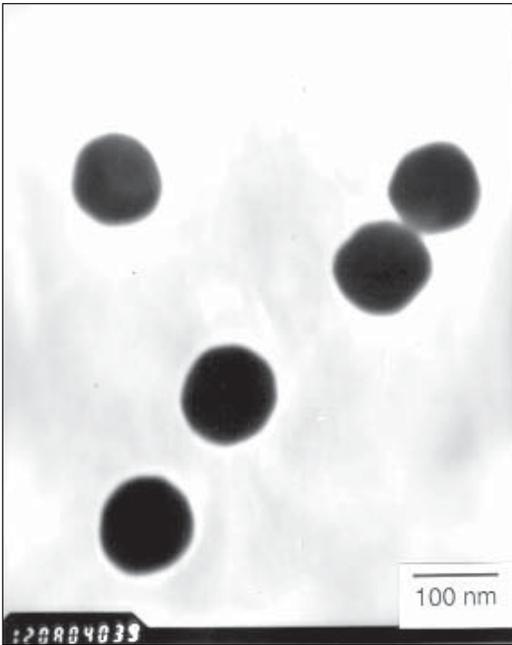


Figure 2. Transmission electron microscope images of 100 nm copper spheres generated by a novel spray pyrolysis method. These particles were developed to test light scattering theories for scattering by particles on surfaces.

DELIVERABLES: Publication of manuscript on the slip correction of nano-size particles based on the DMA. 1Q 2005

5. Resource on Particle Science – Over the past five years, the particle related work has included projects with SEMATECH and particle suppliers to the semiconductor industry. A number of needs by particle related companies were expressed at a NIST particle workshop including redoing the uncertainty assessments of existing particle SRMs and offering a particle sizing calibration service. Providing support for particle needs critical to the semiconductor industry will continue to be a priority.

DELIVERABLES: Provide technical support for the development of improved methods for calibrating surface inspection systems through the SEMI Advanced Wafer Surface Inspection System task force. 2Q 2005

ACCOMPLISHMENTS

- Determined that the electrospray technique can produce an aerosol having characteristics optimal for transferring particles in a liquid suspension onto wafers for particle diameters as small as 25 nm. This significant finding enables improved wafer depositions by reducing the number of contaminant residue particles, the number of doublets, and the amount of residue on the particles.

- Developed a NIST Calibration Facility for sizing monodisperse spheres suspended in water in the size range of 50 nm to 400 nm with an expanded uncertainty of 1.5 % of the peak size. This facility has been used for two customers providing calibration particles to the semiconductor community.

- Coordinated the experimental design and uncertainty analysis with the University of Minnesota for the first measurement of slip correction of particles smaller than 300 nm. These measurements are critical to improving the accuracy of particle size by the DMA in the nanometer size range.

- In collaboration with the University of Maryland, developed a method for generating pure copper spheres with diameters ranging from 100 nm to 200 nm. These spheres, which mimic real-world particles better than polystyrene, were used to validate particle scattering theories in conditions for which models have a higher degree of uncertainty. Measured polarization and intensity of light scattered from the copper spheres and found good agreement with the Bobbert-Vlieger theory for light scattering from a sphere above a surface.

- Developed a method, based upon scattering ellipsometry, for quantifying scatter from two sources and demonstrated its use by characterizing the roughness of both interfaces of an SiO₂/silicon system. This finding establishes the validity of the light scattering models for roughness in a dielectric film, which in turn limits the detection sensitivity of SSIS instruments. The method was also used to characterize scattering from steel surfaces, demonstrating capability to distinguish between scattering from surface roughness and material inhomogeneity. The method was further used to study the scatter from an anti-conformal polymer film, helping to establish the limits of validity of the scattering theory.

- Developed the SCATMECH library of C++ routines for light scattering. Published the SCATMECH library, providing a means for distributing scattering models and polarized light calculations to others. From the time of its public availability in March 2000, over 2500 copies of the library have been downloaded from the web. The Modeled Integrated Scatter Tool (MIST), a Windows application for calculating scatter, was released in June 2004. In July 2005, Version 5 will be released, which contains a theory for scattering of light by a deformed particle on a surface.

■ Extended the theory of scattering of a sphere on a surface to axially-symmetric non-spherical particles. Demonstrated that the scattering by a metal particle on a surface is extremely sensitive to the shape of the particle in the region where the particle contacts the surface. This unusual sensitivity to shape must be considered when light scattering tools classify particles for material and size.

■ Performed a light-scattering-based diameter measurement of the NIST 100 nm PSL sphere standard (SRM 1963) deposited onto a silicon wafer. The measurement results included a thorough assessment of the uncertainties that arise in such measurements. It was found that the uncertainty was dominated by the uncertainty in the shape of the particle on the surface. Results for smaller PSL particles suggest that surface-induced deformation of the particles must be considered.

COLLABORATIONS

Department of Chemical Engineering, University of Maryland, Professor Sheryl H. Ehrman, Validation of Scattering Theory Using Novel Monodisperse Particles.

Department of Mechanical Engineering, University of Minnesota, Professor David Pui, Generation and Measurement of Nanosize Particles.

National Metrology Institute of Japan, Dr. Kensei Ehara, Nanoparticle Metrology.

RECENT PUBLICATIONS

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FRONT-END MATERIALS CHARACTERIZATION

GOALS

To provide industry with new and improved measurements, models, data, and measurement traceability/transfer mechanisms to enable the more useful and more accurate metrology infrastructure needed for select silicon CMOS front-end materials characterization. Major focus is placed on metrology requirements from the 2004 Update to the International Technical Roadmap for Semiconductors (ITRS) expressed as difficult challenges: (1) Structural and elemental analysis at the device level, including SIMS and HRTEM, and (2) Metrology for advanced gate stacks and other thin films. Additional work will be undertaken as possible on additional important thin films and bulk material properties for silicon and other emerging semiconductors.

(1) To improve capabilities for compositional depth profiling, this project defines optimum procedures for ultra-high depth resolution by Secondary Ion Mass Spectrometry (SIMS), develops depth-profiling reference materials needed by U.S. industry, and improves the uncertainty of implant dose measurements by SIMS.

(2) To address needs in composition and thickness measurements for thin films and interfaces, this project develops new optical and physical characterization methods, as well as, characterizes the accuracy and reliability of existing methods. Materials of interest include high- κ and low- κ materials, polymers, silicon-on-insulator (confined silicon), and strained silicon-germanium. High Resolution Transmission Electron Microscopy (HRTEM) is being developed as a chemical tomography tool for determining 3-D elemental distributions in advanced materials.

(3) Determine the work function, band offset, and interfacial structures of high- κ /combinatorial metal electrode stacks systems by implementing a combination of techniques including Scanning Kelvin Probe Microscopy (SKPM), internal photoemission (IPE), backside FTIR, and external photoemission (Soft XPS and Inverse photoemission), and theoretical modeling.

CUSTOMER NEEDS

The Front-End Materials Characterization project addresses key material characterization problems associated with the integrated circuits industry's front-end process, particularly new gate stack pro-

cesses and materials, and metrology for structural and element characterization at the device level, particularly ultra-shallow junctions. Front-end processing requires the growth, deposition, etching, and doping of high quality, uniform, defect-free films. These films may be insulators, conductors, or semiconductors. The 2004 International Technology Roadmap for Semiconductors (ITRS) near-term (through 2009) difficult challenges for front-end processes include: *metrology issues associated with gate dielectrics film thickness and gate stack electrical and materials characterization, introduction of metal gate electrodes with appropriate workfunctions, and metrology issues associated with 2-D dopant profiling. Metrology needs for Thermal/thin films, Doping Technology, SOI, and strained-silicon are discussed in the 2003 International Technology Roadmap for Semiconductors in the Metrology Section and the 2004 Update.*

Since source/drain dopant profiles are a critical factor determining the performance of a transistor, dopant profiling has always been needed by the silicon integrated circuit industry. One-dimensional dopant profiles from SIMS or electrical techniques remain an important process control tool. As transistors are scaled to ever-smaller dimensions, the variation of dopant profiles in two- and three-dimensions also begin to influence device operation. Two- and three-dimensional dopant profiles are now needed to validate models of the processes used to produce ultra-shallow junctions and for accurate device simulations.

SIMS is most likely to provide the solution to precision requirements for 1-D dopant concentration measurements. These goals can be achieved by careful control of SIMS depth-profiling conditions and by developing and making available implant reference materials for common dopant elements.

The ITRS identifies structural and elemental analysis at device dimensions (for example 3-D dopant profiling) as one of the difficult challenges beyond 2009. Offline secondary ion mass spectroscopy has been shown to provide the needed precision for current generations including ultra-shallow junctions. Two- and preferably three-dimensional profiling is essential for achieving future technology generations. Activated dopant profiles and related TCAD modeling and defect

Technical Contacts:

Greg Gillen: SIMS
David Simons: SIMS
John Small: TEM,
X-ray detectors
John Suehle: Electrical
Characterization
Nhan Nguyen: IPE, SE

profiles are necessary for developing new doping technology. The ITRS requirements are for at-line 2-D dopant profile concentration measurements with spatial resolution of 4.1 nm and precision of 4 % in 2004, increasing to spatial resolution of 2.8 nm and 2 % precision for the 2010 through 2018 timeframe. Complete specifications are given for the short term in Table 119a on page 12, and for the long term in Table 119b on page 13 of the 2004 Update to the Metrology section.

The interface of high- κ gate dielectrics with the metal electrode presents a most critical challenge to the development of a gate stack that meets specifications required by the semiconductor industry. Numerous challenges for these materials exist:

- Defects, as well as interfacial reaction products, at the metal gate electrode/high- κ gate dielectric interface may impact the work function of the gate electrode and the band offset between the gate dielectric and the electrode.
- The thermal stability of the interface strongly impacts the minimum achievable EOT.
- A wide range of binary and ternary compositions for the metal gate electrode are possible.
- A variety of conflicting measurements of the work function and band offsets exist.

TECHNICAL STRATEGY

The 2003 ITRS expressed as difficult challenges: (1) structural and elemental analysis at the device level, including SIMS and HRTEM, and (2) metrology for advanced gate stacks and other thin films. Our focus areas include development of refined metrology methods and standards for SIMS and TEM, developing improved X-ray detection capabilities for SEMs and electron microprobes, and the characterization of high- κ /metal gate interfaces, including band offsets and barrier heights.

STRUCTURAL AND ELEMENTAL ANALYSIS AT THE DEVICE LEVEL, INCLUDING SIMS AND HRTEM

Secondary ion mass spectrometry (SIMS) has demonstrated the capability to meet the ITRS dopant profiling requirements for B, As, and P. However, the detailed analytical protocols required to achieve these goals have not been completely specified. We have organized an interna-

tional round robin study through ISO committee TC201 to investigate the parameters that must be controlled to make highly repeatable dose measurements of As with SIMS instruments. In addition to improved repeatability for dopants, the ITRS roadmap also requires increased SIMS detection limits for trace metal and organic contamination analysis of semiconductor devices. We are also working on novel methods to enhance detection limits for common metal contaminants by increasing the ionization efficiency during the SIMS sputtering process.

DELIVERABLE: Evaluate results of international round robin of arsenic dose determination by SIMS. Develop improved methods for high sensitivity analysis of trace surface metals and impurities. 1Q 2005

Analysis of trace metal and organic contamination on silicon surfaces is a high priority of the ITRS roadmap. To utilize effectively tools such as secondary ion mass spectrometry for trace contamination on silicon surfaces, suitable trace standards must be developed. Over the past 20 years, piezoelectric drop-on-demand ink-jet printing has evolved into a precision microdispensing technology with a diverse range of applications. Examples of applications include desktop color printers, printing and synthesis of DNA arrays and printing of molten solder for use as electrical interconnects on integrated circuits. We are exploring the possibility of using ink-jet technology to print elemental and organic contamination standards on silicon. Piezoelectric printers are capable of printing single microdrops of fluid at the rate of thousands of drops per second. Each drop contains a known concentration of the material of interest. Large concentration ranges are possible simply by varying the number of drops printed. Our first attempts will explore ink-jet printing of organic test dyes on silicon with subsequent characterization by SIMS (see Fig. 1). Once standard operating procedures are developed for ink-jet printing, it should be feasible to produce standards, (both organic and trace metal) for quality control and calibration of a variety of analytical techniques including SIMS, XPS, AES, EPMA, TXRF and others.

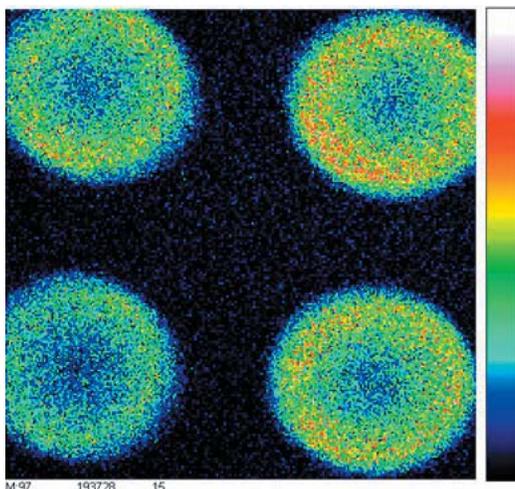


Figure 1. Inkjet Droplets of an organic dye on silicon substrate. NIST TOF-SIMS image, field-of-view 500 μm .

DELIVERABLE: Determine feasibility of ink jet printing of trace organic and metals onto silicon. 2Q 2005

As integrated circuit dimensions shrink to the sub-micrometer regime, there is continued need for accurate and quantitative dopant depth profiling with ultra-high-depth resolution. To probe shallow dopant profiles in Si and other materials, SIMS instruments typically utilize very low energy primary ion beams to bombard the sample surface. In this case, it is difficult to obtain a well-focused and high current density beam, especially in a magnetic sector SIMS instrument. Recently, there has been growing interest in using molecular ion beams for depth profiling. When a molecular primary ion beam impacts the surface, it dissociates into its constituent atoms with each atom retaining a fraction of the initial energy of the cluster. This process can lead to impact energies on the order of a few 10's of electron volts and a corresponding reduction the depth of penetration of the primary ion. This process may potentially allow for ultra high resolution depth profiling. In this project, we will utilize a new C_{60}^+ cluster primary ion beam source at NIST to sputter depth profile Si, GaAs, SiC, and multiple delta-layers test materials.

Some thin-film materials such as metals do not sputter as uniformly as silicon under ion bombardment. In these cases, the achievable depth resolution is limited not by the penetration depth of the primary ion but by the topography induced by the sputtering process itself. We will also explore the use of cluster bombardment SIMS for reduction

of sputter induced topography in metal films. This approach will be applied to study depth profiling analysis of gold diffusion in copper and the depth distribution of blanket metals films (copper metalization on silicon).

DELIVERABLES: Improve SIMS depth profile resolution in silicon using large cluster ion beam analysis. Explore the feasibility of reducing sputter induced topography in metal films using cluster SIMS. 3Q 2005

SiGe THIN FILM COMPOSITIONAL STANDARDS

After the need for SiGe compositional standards was discussed at the 14th Annual Workshop on Secondary Ion Mass Spectroscopy (SIMS) in 2001, NIST contacted fabrication and analytical laboratory facilities to develop a reference material using an interactive approach. The typical time frame for an NIST SRM is many years to develop, characterize, and bring to market. The need for SiGe standards was seen as an immediate need, because the current calibration method of Rutherford Backscattering is only accurate from 5 % to 10 % relative and this is insufficient for reliable device production. NIST is collaborating with several semiconductor facilities and analytical laboratories to develop a suite of SiGe compositional standard films. Using an interactive data collection mechanism with collaborators and publishing the data on the web, a faster approach to standard materials production is being developed. This will allow materials to be used and compared even while reference data are being developed.

DELIVERABLE: Develop a suite of SiGe compositional standards to allow accurate and reproducible SiGe thin film production. 4Q 2004

ACCOMPLISHMENTS

IMPLEMENTATION OF C_{60}^+ CLUSTER ION SIMS CAPABILITY

- Previous efforts with cluster ion sources used for analyzing both organic and inorganic materials have been very successful. Minimization of beam-induced damage in organic materials has allowed depth profiling of polymers such as photoresists and enhanced ion yields for high-molecular weight fragments. Inorganic material analysis has benefitted in the area of ultra-shallow depth-profiling as well as for analysis of some particularly difficult systems such as metal multi-layers stacks. We have investigated the use of a commercially available C_{60}^+ ion source on the NIST

magnetic sector SIMS instrument (see Fig. 2). We have produced stable ion beams of C_{60}^+ and C_{60}^{2+} with typical currents approaching 20 nA under conditions that allow for several hundred hours of operation. The beam can be focused into a spot size of $\sim 1 \mu\text{m}$ allowing micrometer spatial scale mapping of patterned wafers. Optimal experimental conditions have been defined to allow for depth profiling analysis of silicon wafer samples, delta doped structures and metal multilayers.

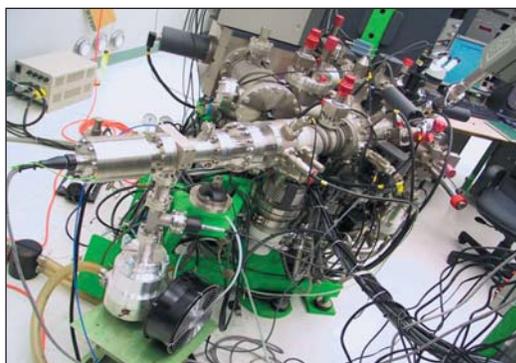


Figure 2. C_{60} ion source mounted on NIST magnetic sector SIMS instrument.

DEPTH PROFILING OF ORGANIC OVERLAYERS USING SIMS

Organic photoresists and low- κ dielectric materials are key components for front end semiconductor processing. There is also a growing interest in the use of organic semiconductor materials for organic light emitting diodes and organic thin film transistors. In anticipation of a growing need for metrology tools to characterize these types of materials, we are developing new approaches to characterize the chemical composition and in-depth distribution of organic thin films on silicon. Typically, the use of ion beam sputtering techniques, such as secondary ion mass spectrometry (SIMS), results in extensive chemical degradation of organic thin films such as photoresists or organic light emitting diodes. However, we have found that cluster primary ion bombardment SIMS can minimize this degradation allowing for intact characteristic ions to be obtained throughout the depth of the film. Furthermore, it appears that analyzing these organic materials at cryogenic temperatures provides further reduction in beam-induced damage. In this project, an SF_5^+ polyatomic primary ion source was used to SIMS depth profile Poly(methyl methacrylate) (PMMA) photo resist at a series of temperatures from 198 K to 398 K where the pri-

mary glass transition for PMMA occurs at 378 K. The depth profile characteristics (*e.g.*, interface widths, sputter rates, damage cross sections, and overall secondary ion stability) were monitored as a function of temperature. It was found that at low temperatures, the secondary ion stability increased considerably. In addition, the interfacial widths were significantly lower. Examples of this increased stability at low temperatures are illustrated in Fig. 3 for the PMMA fragment at $m/z = 69$. Corresponding AFM images indicated that there was also decreased sputter-induced topography formation at these lower temperatures. Higher temperatures were typically correlated with increased sputter rates. However the improvements in interfacial widths and overall secondary ion stability were not as prevalent as was observed at low temperatures. The importance of the glass transition temperature (T_g) on the depth profile characteristics was also apparent. The results of this study demonstrate that it is possible to monitor the chemical composition of photoresist thin layers on silicon by analyzing the samples at cryogenic temperatures.

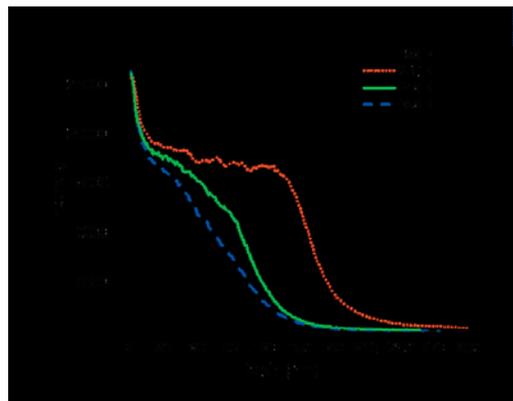


Figure 3. Positive ion intensities of characteristic fingerprint ion of PMMA $m/z = 69$ plotted as a function of depth for a PMMA film on Si ($\approx 160 \text{ nm}$): measured at varying temperatures (at and below room temperature).

COMPOSITION DEPTH PROFILING OF FE/Ni AND Pt/Co MULTILAYERS USING SIMS

Secondary ion mass spectrometry (SIMS) is a powerful technique for the in-depth analysis of solid materials. However, SIMS is difficult to apply for the quantitative analysis of major components due to severe matrix effects. Therefore SIMS is more commonly used for

the quantitative analysis of minor impurities. In this work, a C_{60}^+ ion beam was studied as a sputtering source for the quantitative analysis of binary alloy films and the quantitative depth profiling of multilayer films. Fe/Ni and Pt/Co multilayer thin films were grown on Si (100) wafers by ion beam sputter deposition. Fe-Ni and Pt-Co alloy films were also grown for quantitative surface analysis of major components. The compositions of Fe-Ni alloy thin films were certified by an isotope dilution method using inductively coupled plasma-mass spectrometry (ICP-MS) and those of Pt-Co alloys were certified by ICP-optical emission spectroscopy (OES). Calibration curves were derived from linear fits between the nominal compositions and the SIMS compositions calculated using relative sensitivity factors from reference alloy films ($Fe_{51}Ni_{49}$, $Pt_{40}Co_{60}$) as shown in Figure 4. SIMS depth profiling was performed with a magnetic sector SIMS system using 14.5 keV impact energy C_{60}^+ ions and negative ion detection. The slope of 1.034 and an offset value of -1.89 % in the calibrated SIMS compositions of Fe-Ni alloy films showed a good correlation with nominal compositions. No interface artifacts were found in a depth profile of an Fe/Ni multilayer. However, for Pt-Co alloy films, the calibration line slope was 0.898 and the offset value was 3.04 % due to matrix effects.

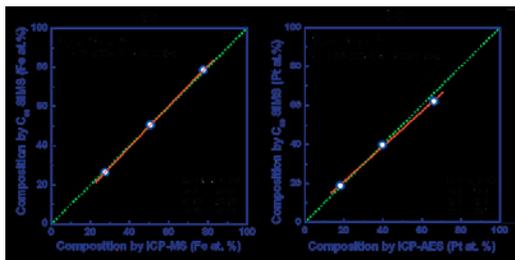


Figure 4. Calibration curves of (a) Fe-Ni and (b) Pt-Co alloy films derived from linear fits using relative sensitivity factors from reference alloy films ($Fe_{51}Ni_{49}$, $Pt_{40}Co_{60}$)

SiGe Compositional Standards – SiGe specimens cut from single-crystal boules normal to the growth axis were obtained from Virginia Semiconductor. The nominal compositions were 3.5 at. % Ge in Si, 6.5 at. % Ge in Si, and 14 at. % Ge in Si. The wafers were evaluated with the electron probe microanalyzer (EPMA) for micro- and macroheterogeneity for use as primary standards for characterization of SiGe thin

films on Si that are needed by the microelectronics industry as reference standards. The specimens were rigorously tested with wavelength dispersive spectrometers (WDS) using multiple point, multiple sample, and duplicate data acquisitions. On each specimen, two 40-point traverses normal to one another were prepared at two randomly selected locations with steps of 2 μm or 5 μm between points. Such traverses are routinely prepared during heterogeneity testing to determine if distinct phases across short distances (5 μm to 100 μm) within specimens are present. In addition random sampling tests on each specimen were run to evaluate the overall specimen heterogeneity. The expanded uncertainties (3σ) in percent mass fraction determined from data for each specimen ranged from 1–2 % in $Si_{.86}Ge_{.14}$ to as high as 15 % in $Si_{.965}Ge_{.035}$. The $Si_{.86}Ge_{.14}$ was chosen as the primary standard for the SiGe films on Si due to the low heterogeneity of this material for which the maximum expanded uncertainty due to heterogeneity is no greater than 1.5 % relative mass fraction for Ge and considerably less for Si.

Round-Robin Study of Arsenic Implant Dose Measurement in Silicon by SIMS – An international round-robin study was undertaken under the auspices of SIMS subcommittee SC 6 of International Organization for Standardization Technical Committee TC 201 on Surface Chemical Analysis. The purpose of the study was to determine the best analytical conditions and the level of interlaboratory agreement for the determination of the implantation dose of arsenic in silicon by SIMS. Motivations for this study were: (a) the relatively poor interlaboratory agreement that was observed in a previous round-robin study before a certified reference material had become available; (b) the observation that the use of Si_3^- as a matrix species combined with AsSi⁻ detection may result in improved measurement repeatability compared with Si_2^- ; and (c) the observation that point-by-point normalization can extend the linearity of SIMS response for arsenic in silicon beyond $1 \times 10^{16}/cm^2$.

Fifteen SIMS laboratories participated in this study, as well as two laboratories that performed Low energy Electron-induced X-ray Emission Spectrometry (LEXES) and one that made measurements by Instrumental Neutron Activation Analysis (INAA). The labs were asked to determine the implanted arsenic doses in three unknown samples using as a comparator Standard

Reference Material 2134, with a certified dose of 7.33×10^{14} atoms/cm². The use of a common reference material by all laboratories resulted in much better interlaboratory agreement than was seen in the previous round-robin that lacked a common comparator. The relative standard deviation among laboratories was less than 4 % for the medium-dose sample, and somewhat larger for the low- and high-dose samples (see Fig. 5). The high-dose sample showed a significant difference between point-by-point and average matrix normalization because the matrix signal decreased in the vicinity of the implant peak, as previously observed. The average dose from point-by-point normalization was in close agreement with that determined by INAA, indicating that the SIMS relative sensitivity factor approach is valid for arsenic concentrations in silicon as high as 4 atom percent.

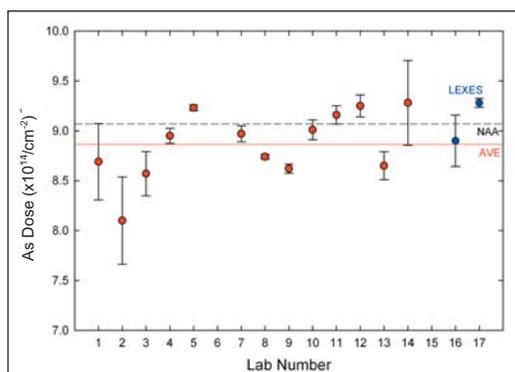


Figure 5. As round robin results for medium dose sample with point-by-point normalization to matrix signal. SIMS lab results shown in red with error bars indicating within-lab repeatability. Relative standard deviation among labs is 3.9 %. Also shown are LEXES results (blue dots), average of SIMS lab results (red line), and NAA result (black dashed line).

Enhanced Performance of Microcalorimeter X-ray Detector – We have previously reported improvements to stability of the microcalorimeter x-ray detector by stabilizing the temperature of the detector to 1 part in 1000. This effort enabled the collection of X-ray spectra over periods of hours which showed resolutions on the order of 12 eV and drifts less than 2 eV. Recently, CSTL scientists working with NIST Boulder have identified the previously observed problem of abrupt jumps in the spectral lines as due to the high-speed pulse feedback loop losing lock and relocking on a different flux quantum. We are investigating hardware improvements to eliminate this problem. We have received from NIST boulder an upgraded microcalorimeter thermometer

including a new Nb superconducting shield. As a result of these modifications, the upgraded CSTL microcalorimeter has obtained a resolution at Mn K α (5.9keV) of 4.39 ± 0.15 eV (see Fig. 6).

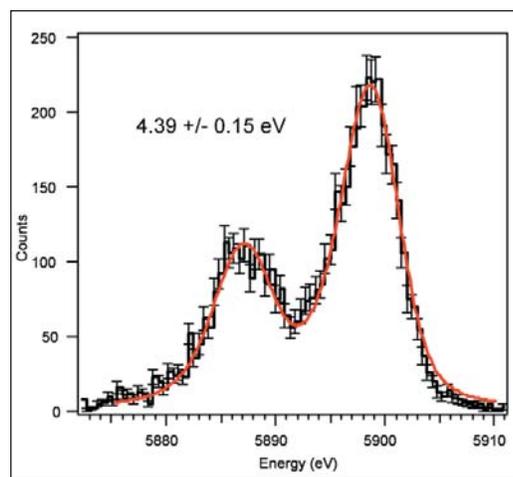


Figure 6. X-ray spectrum taken with the upgraded CSTL microcalorimeter of Mn K α 2 (5.887 keV) and Mn K α 1 (5.898 keV) peaks showing 4.39 eV resolution. Note: No software drift correction has been applied.

Methods for depth-sensitive imaging by Scanning Transmission Electron Microscopy – Recently, researchers have begun to explore the possibility of chemical tomography in the Analytical Electron Microscope (AEM), the determination of 3-D elemental distributions in the sample based on tilt-series of energy-filtered TEM (EFTEM) elemental maps and high-angle annular dark field (HAADF) STEM structural data. One of the chief barriers to this work is that for thicker samples EFTEM maps can show non-monotonic relationships between the observed signal in the 2-D tilt images and the concentration of the analyte in that projected pixel. This violates the projection requirement, a key assumption in many 3-D reconstruction algorithms. Because of this, most successful work to date has been limited to systems that (at least approximately) meet this requirement and do not exhibit the full complexity of interactions possible in the AEM. To get past this hurdle, it is necessary to construct new models for 3-D reconstruction in the AEM that are not based on X-ray tomography predecessors and that explicitly account for effects such as beam spreading, multiple scattering, and through-sample self-absorption. Following the lead of the medical imaging community, one useful step toward the creation of effective 3-D re-

construction algorithms is the analysis of synthetic datasets of phantoms with known properties. Figures 7-1, 7-2, and 7-3 show the results from 3-D Monte Carlo simulations of three cylinders (Cu, Al, and SiO₂), each 600 nm in diameter and 1 μm long. The electron beam (purple line) was rastered over 32 pixels parallel to the x-axis and the resulting X-ray spectra at the XEDS detector were calculated, resulting in spectrum-line profiles for each tilt from 0 degrees to 180 degrees. From the 5,760 spectra calculated, chemical sinograms were extracted by summing the intensities in 130 eV wide windows over the Cu Kα, O Kα, and Al Kα peaks and displaying them as the channels of an RGB image (Fig. 7-4). These data display both beam broadening and pronounced self-absorption effects, but not Bragg scattering or dynamical interactions, two other effects that plague 3-D AEM.

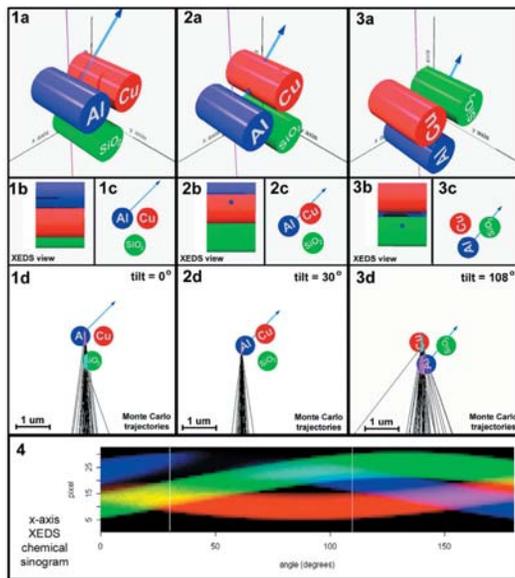


Figure 7. 1(a) 0 degree tilt perspective view of AEM cylinder phantoms (600 nm diameter by 1 μm long); blue arrow points to XEDS detector. (b) same phantoms as seen by XEDS detector. (c) view along tilt axis. (d) 300 keV Monte Carlo electron trajectories. 100 electrons displayed at 1 of 32 horizontal beam raster locations. Figures 2 & 3 same as Figure 1 but sample tilted to 30 degrees and 108 degrees respectively. Figure 4. “Chemical sinogram” displaying XEDS line intensities vs. sample tilt angle (horizontal axis) and x-axis beam raster position (vertical axis). Red is Cu Kα, green is O Kα, and blue is Al Kα. Vertical white lines denote 30 degrees and 108 degrees. Note attenuation of Al Kα by Cu cylinder near 30 degree tilt.

CHARACTERIZATION OF THE HIGH-κ/METAL GATE INTERFACE

Measurement of the barrier height and determination of the band structure is not straightforward. Recent work by numerous researchers has shown that the measured band offset between a metal gate electrode and high-κ gate dielectric is dependent on numerous factors including composition, structure, and thickness of both the metal gate electrode and the high-κ dielectric. Because of limitations associated with any single technique, we believe that determination of band offsets and work functions requires the use of an array of techniques and the broad expertise available at NIST. We will focus our efforts on the following measurements: standard Capacitance-Voltage (C-V) and tunneling current-voltage (I-V) measurements, internal photoemission (IPE), scanning kelvin probe microscopy (SKPM), and soft X-ray and inverse photoelectron spectroscopy (in collaboration with Rutgers University).

Internal Photo Emission (IPE) – An HfAlO film on a p-type silicon substrate was fabricated by Atomic Layer Chemical Vapor Deposition (ALCVD). Targeted film composition, designated by 25 [5 HO : 1AO], was deposited by 5 cycles of H₂O/TMA precursor followed by 1 cycle of H₂O/HfCl₄ precursor and repeated 25 times. The film thickness of 93 Å and the optical bandgaps E_g of 5.76 eV were obtained from modeling the VUV-SE data. MOS capacitors were fabricated for the film by evaporation of 13 nm thick Al electrodes of 0.023 mm² size. IPE measurement was performed in the photon energy range from 1.5 eV to 6.0 eV. The quantum yield (Y) was obtained by normalizing the measured currents to the incidence flux. The spectral threshold was determined by extrapolating Y^{1/2} plot to zero yield. As a result, Fig. 8a shows the negatively biased IPE spectra from which Φ_e (Al), the barrier threshold from the Fermi level to the bottom of the conduction band of HfAlO, was determined to be ~2.4 eV. When positively biased as shown in Fig. 8b, the IPE spectra yield a barrier height of ~3.2 eV corresponding to the threshold from the silicon valence band to the bottom of the HfAlO conduction band. Both of these values agree with the published data within 0.1 eV. Also a slight feature in the positive bias spectra (Fig. 8b) was observed at ~5.7 eV which is attributed to the optical excitation in the HfAlO film; it is the same as the optical bandgap E_g measured by VUV-SE.

DELIVERABLES: Demonstrated the performance of IPE tool on metal/high- κ /Si structure to determined band offsets and compare with values in literature. 1Q 2005.

DELIVERABLES: Complete measurements of a set of HfO₂ and HfSiO using vacuum ultraviolet spectroscopic ellipsometry (VUV-SE), X-ray diffraction (XRD), Atomic force microscopy (AFM), and FTIR Grazing Angle Attenuated Total Reflection (FTIR-GATR). 3Q 2005.

DELIVERABLES: Determine Optical constants for Ti-Ni-Pt ternary system using vacuum ultraviolet spectroscopic ellipsometry. 3Q 2005.

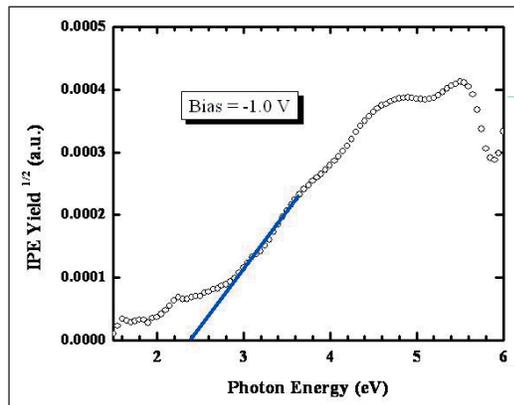


Figure 8a. IPE spectra from a negatively IPE spectra for HfAlO/Al system. Φ_e (Al), the barrier threshold from the Fermi level to the bottom of the conduction band of HfAlO, was determined to be ~ 2.4 eV.

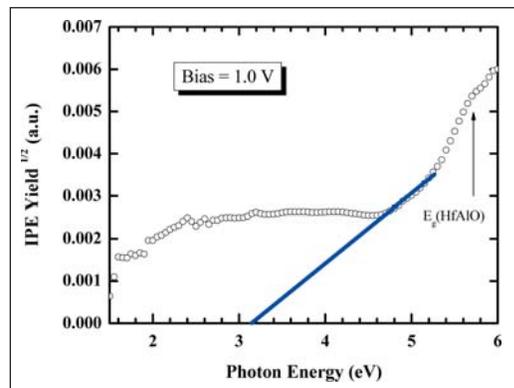


Figure 8b. IPE spectra for a positively biased HfAlO/Al system. A feature was observed at ≈ 5.7 eV which is attributed to the optical excitation in the HfAlO film and is the same as the optical bandgap E_g measured by VUV-SE.

Scanning Kelvin Probe Microscopy (SKPM)

– Kelvin probe is a non-invasive, non-contact vibrating capacitor technique, which measures the voltage between a vibrating micro-electrode and a conducting or semiconducting sample with millivolt resolution. The surface potential measurement contains information on the sample's work function. Baseline measurements of well known inert metals such as gold and platinum (on SiO₂/Si) will validate the procedures and interpretation of the scanning Kelvin probe measurements. Unlike C-V, I-V, or IPE, Kelvin probe measures the surface work function of the metal gate electrode. This surface work function may or may not directly relate to the interfacial band offsets. However, a change in the surface work function will provide important clues necessary to understand the band offsets and final device threshold voltage. We are developing a mask set that will permit the comparison of Kelvin probe with standard C-V on a given wafer. We also explored surface treatments necessary to obtain a valid metal work function. The optimization of sample preparation and the careful characterization of the SKPM tip work function have yielded excellent agreement of seven sample metal work functions to literature values as shown in Fig. 9.

DELIVERABLES: Compare metal work functions obtained from SKPM with measurements made by CV technique on a ternary metal/high- κ system fabricated by combinatorial techniques. 3Q 2005.

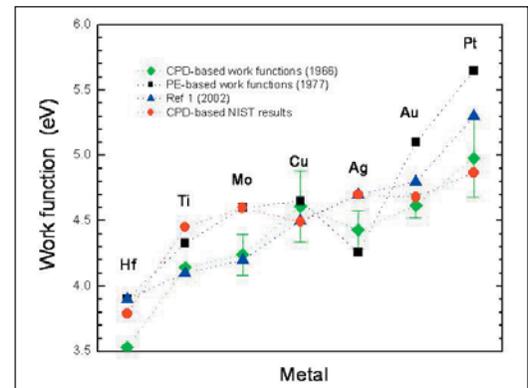


Figure 9. Comparison of metal work function obtained from the NIST SKPM with literature values. The agreement is good after the optimization of sample preparation and analysis procedure.

COLLABORATIONS

FM Technologies – High brightness oxygen ion source for 2-D dopant profiling by SIMS

International SEMATECH, Joe Bennett – Thin oxide depth-profiling by SIMS, backside depth profiling of patterned PMOS wafers

Ionoptika – Development of a C_{60}^+ primary ion source for advanced semiconductor technology

MicroFAB Inc – Advanced inkjet printing technology for deposition of trace metal standards on silicon surfaces

Peabody Scientific – Ion source development for Semiconductor SIMS

SEMATECH, Characterization of metal gate/high- κ systems.

University of Maryland, College Park, Ultra-thin gate oxide reliability.

University of Minnesota, Alternate Gate Dielectrics.

MSEL, Characterization of metal gate/high- κ systems.

IBM, Electrical characterization of high- κ systems.

Texas Instruments, Electrical characterization of high- κ systems.

Rutgers University, Characterization of metal gate/high- κ systems.

Yale University, Electrical characterization of high- κ systems.

RECENT PUBLICATIONS

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K.J. Kim, D.W. Moon, P. Chi and D. Simons, “Development and Applications of Multiple Delta-Layer Reference Materials for Semiconductor Analysis,” *J. Surf. Anal.*, accepted for publication.

D.S. Simons, P.H. Chi and K.J. Kim, “Quantitative Measurement of Arsenic Implant Dose by SIMS,” *J. Surf. Anal.*, accepted for publication.

K.J. Kim, D.W. Moon, P. Chi and D. Simons, “Development of B-doped Si Multiple Delta-Layer Reference Materials for SIMS Profiling,” *Surf. Interface Anal.*, submitted for publication.

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CHEMICAL METROLOGY OF MATERIALS

GOALS

To provide industry with new and improved measurements, models, data and measurement traceability/transfer mechanisms to enable the more useful and more accurate metrology infrastructure needed for select silicon CMOS front-end materials characterization. Major focus is placed on metrology requirements from the 2004 update of the 2003 International Technical Roadmap for Semiconductors expressed as difficult challenges: (1) Measurement of complex materials stacks and interfacial properties including physical and electrical properties, (2) Structural and elemental analysis at device dimensions, and (3) Nondestructive production worthy wafer and mask level microscopy for critical dimension measurement for 3-D structures, overlay, defect detection, and analysis.

CUSTOMER NEEDS

Materials with high dielectric constants such as HfO_2 , ZrO_2 , HfSiO_4 , and ZrSiO_4 are candidate "high- κ " gate-dielectric materials in future semiconductor devices. These materials can have larger physical thicknesses than SiO_2 in order that the equivalent oxide thickness (corresponding to an SiO_2 dielectric) can be reduced. For devices produced between 2006 and 2009, the equivalent oxide thickness will be between 0.7 nm and 1 nm for high-performance logic and between 1.6 nm and 1.9 nm for low-operating-power logic. These thicknesses need to be measured with an uncertainty (3σ) of $\pm 4\%$.

X-ray photoelectron spectroscopy (XPS) is a frequently used technique for characterizing thin-film materials and for measuring thicknesses of gate-oxide films. Commercial tools are now available for such XPS measurements on 300 mm wafers. The material parameter needed for measuring thicknesses of overlayer films by XPS is the effective attenuation length (EAL). EAL data are needed for candidate high- κ materials for common XPS measurement conditions.

Auger-electron spectroscopy (AES) is extensively used for the characterization of defects on wafers. Although commercial AES instruments have a lateral resolution of about 10 nm, the detected Auger signal will typically originate from much larger regions due to the effects of backscattered electrons. Part of the detected signal arises from ionizations created by the primary beam while

another part comes from ionizations by backscattered electrons. An analyst often needs to make a compromise between making AES measurements at a relatively high beam energy of 25 keV (to optimize the lateral resolution) and making these measurements at much lower beam energies (to reduce the fraction of the detected Auger signal due to backscattered electrons or to reduce charging). Data are needed for the dependence of the analytical area on beam energy for common analytical situations. Such data require more reliable information on electron stopping powers over a wide range of electron energies (typically 100 eV to 30 keV) so that calculations of backscattering factors and analytical areas can be made by efficient Monte Carlo simulations.

TECHNICAL STRATEGY

Calculation of Electron Effective Attenuation lengths – NIST is developing a new database (SRD 100) Simulation of Electron Spectra for Surface Analysis (SESSA) to be used for AES and XPS analyses of thin-film structures. SESSA can be used for two main applications. First, data are provided for many parameters needed in quantitative AES and XPS (differential inverse inelastic mean free paths, total inelastic mean free paths, differential elastic-scattering cross sections, total elastic-scattering cross sections, transport cross sections, photoelectric cross sections, photoelectric asymmetry parameters, electron-impact ionization cross sections, photoelectron line shapes, Auger-electron line shapes, fluorescence yields, and Auger-electron backscattering factors). Second, Auger-electron and photoelectron spectra can be simulated for layered samples. The simulated spectra, for layer compositions and thicknesses specified by the user, can be compared with measured spectra. The layer compositions and thicknesses can then be adjusted to find maximum consistency between simulated and measured spectra. In this way, AES and XPS can provide more detailed characterization of multilayer thin-film materials.

SESSA is being used to compute EALs for thin films of HfO_2 , ZrO_2 , HfSiO_4 , and ZrSiO_4 on a Si substrate. These EALs are determined as a function of film thickness and photoelectron emission angle (*i.e.*, to simulate the effects of tilting the sample in so-called angle-resolved XPS). The calculations were made for excitation by $\text{Al K}\alpha$ X-rays on an XPS instrument with a fixed angle

Technical Contact:
C. J. Powell

of 54 degrees between the X-ray source and the analyzer axis, a common experimental configuration.

DELIVERABLES: Determine electron effective attenuation lengths for XPS thickness measurements of candidate high- κ gate-dielectric materials. 2Q 2005

Calculation of Electron Stopping Powers – Electron stopping powers are being calculated for groups of elemental solids and selected compounds for electron energies between 100 eV and 30 keV. These calculations are based on the same algorithm as that used successfully to compute electron inelastic mean free paths.

A simple equation developed by Bethe has frequently been utilized to describe the dependence of the stopping power on electron energy and material parameters. Although it is known that this equation is invalid for electron energies lower than about 10 keV, the Bethe stopping-power equation has often been used in Monte Carlo simulations of electron transport in solids on account of its analytical simplicity. Efforts are currently underway to fit alternative expressions to the new set of calculated electron stopping powers.

DELIVERABLES: Test candidate analytical expressions for electron stopping powers. 4Q 2005

ACCOMPLISHMENTS

■ **Calculations of Electron Effective Attenuation Lengths** – A new NIST database for the Simulation of Electron Spectra for Surface Analysis (SESSA) has been developed for applications in AES and XPS. Electron effective attenuation lengths (EALs) were computed from SESSA for XPS experiments in which the attenuation of substrate Si 2p photoelectrons is measured through thin overlayer films of HfO₂, ZrO₂, HfSiO₄, and ZrSiO₄ as a function of film thickness and photoelectron emission angle. These EALs were compared to similar values obtained from the NIST Electron Effective-Attenuation-Length Database (SRD 82). Generally good agreement was found between corresponding EAL values, but there were differences for film thicknesses less than the inelastic mean free path of the photoelectrons in the overlayer film. These differences are due to a simplifying approximation in the algorithm used to compute EALs in SRD 82. SESSA, with realistic cross sections for elastic and inelastic scattering in the film and substrate materials, is believed to provide more accurate EALs than SRD 82 for thin-film thickness measurements

by XPS, particularly in applications where the film and substrate have different electron-scattering properties (such as the high- κ materials considered here).

■ **Calculations of Electron Stopping Powers** – Electron stopping powers (SPs) were calculated for ten elemental solids (Al, Si, Cr, Ni, Cu, Ge, Pd, Ag, Pt, and Au). These calculations were made for electron energies between 100 eV and 30 keV. The calculated SPs were compared with measured values, with results of other calculations, with the non-relativistic Bethe SP equation, and with an empirical modification of the Bethe equation. Generally satisfactory agreement of the calculated SPs was found with measured values for some solids (Ni, Cu, Pd, and Pt) and limited agreement for others (Al, Si, Cr, Ge, and Ag). There was similar agreement between the calculated SPs and some results of others, while there was some disagreement in other cases. The calculated SPs agreed with values from the Bethe equation only for energies above 10 keV (for $Z \leq 32$) and about 30 keV for $Z \geq 46$. Satisfactory agreement was found between the SPs and values from an empirical modification of the Bethe equation for Al, Si, Cr, Ni (for energies above 1 keV), Cu, and Ge but there was less agreement for Pd and appreciable disagreement for Au. As an example, Fig. 1 shows the recent NIST results (designated

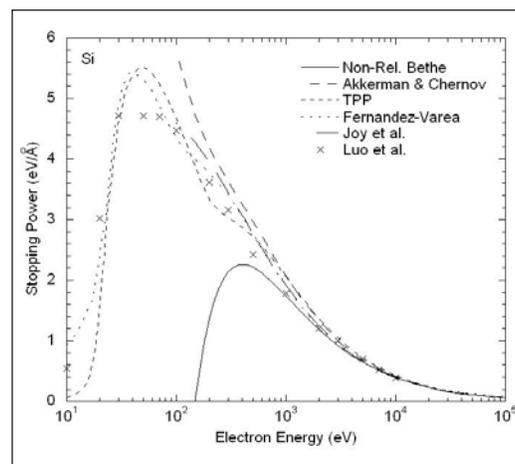


Figure 1. Stopping power for silicon as a function of electron energy. The short-dashed line designated TPP denotes the NIST calculated stopping powers; the dashed lines designated Akkerman and Chernov show results of other calculations; the solid line shows results from the non-relativistic Bethe equation; the long-dashed line denoted Joy et al. shows results from an empirical modification of the Bethe equation; and the symbols denoted Luo et al. show experimental results of Luo et al.

TPP) together with SP data from other sources. In addition, an analysis of the calculated SPs for Al was made to show why the Bethe equation is not expected to be valid unless the electron energy is larger than about 10 keV.

COLLABORATIONS

Institute of General Physics, Technical University of Vienna, Prof. Werner and Mr. Smekal, development of the new NIST database for simulation of electron spectra for surface analysis by AES and XPS, and calculation of electron effective attenuation lengths for XPS measurements of thicknesses of high- κ gate dielectrics on silicon.

Institute of Physical Chemistry (Warsaw), Dr. Jablonski, calculations of electron backscattering factors (for scanning Auger microscopy), and analyses of electron stopping powers.

National Institute for Materials Science (Tsukuba), Dr. Tanuma, calculations of electron stopping powers, and analyses of stopping-power data.

NIST, 841, Dr. D. R. Penn – calculations of electron stopping powers.

RECENT PUBLICATIONS

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