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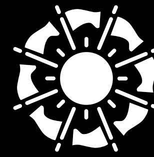

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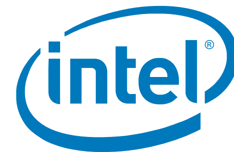

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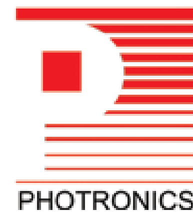

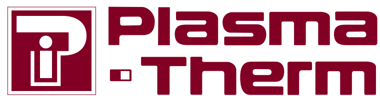

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Dale E. Newbury
National Institute of Standards and Technology

S. Frank Platek
U.S. Food and Drug Administration

Tim K. Maugel
Univ. of Maryland, College Park

9636-2, Session 1

Advances in FIB ex situ lift out specimen preparation *(Invited Paper)*

Lucille A. Giannuzzi, EXpressLO LLC (United States)

The Ga+ focused ion beam (FIB) instrument combined with a scanning electron microscope (SEM) creates a dual platform instrument that enables site specific specimen preparation for 2D and 3D analysis and nanoprototyping. FIB techniques are particularly useful for preparing thin specimens for transmission electron microscopy (TEM) and examples of FIB/TEM applied to a range of material systems will be given. Extraction of FIB specimens from bulk materials require lift out techniques to be employed. The ex situ lift out (EXLO) technique is historically the first lift out technique developed. EXLO is well known for its ease, speed, and reproducibility, and is perfectly suited for manipulation of electron transparent specimens for S/TEM or thicker specimens for surface science analysis or analytical scanning electron microscopy. Adhesion forces secure the specimen to its supporting surface. The EXLO manipulation capabilities also aid in specimen preparation for particles and may be used for preparing fibers or other particulate materials. EXLO is also perfectly suited for manipulation of electron transparent specimens to MEMS carrier devices used for in situ TEM holders. The development of a new grid design and technique allows EXLO and manipulation without needing a thin film support. The specimen is lifted out and manipulated directly to a slotted grid surface such that the specimen may be directly analyzed and/or further FIB milled, broad beam ion milled or plasma cleaned. Using this new grid design, a specimen can also be easily manipulated into a “backside” orientation which avoids curtaining artifacts after further FIB milling. Large Xe+ plasma FIB specimens can be lifted out via the EXpressLO™ technique. Lift out applications will be presented, adhesions forces will be discussed, and various lift out techniques for FIB prepared specimens will be shown.

9636-3, Session 2

Status of accurate three-dimensional SEM measurements at the nanometer scale *(Invited Paper)*

András E. Vladár, Vipin Tondare, John S. Villarrubia, Michael T. Postek, National Institute of Standards and Technology (United States)

With the advances of nanotechnology, there is an increasing need for three-dimensional (3D) metrology with atomic-scale accuracy. Since the unique properties of nanodevices and nanoscale structures are inherently dependent on their dimensions, more accurate metrology methods are needed for research, development, and manufacturing process control.

The scanning electron microscope (SEM) is used extensively in research, development and manufacturing. Today’s best instruments can achieve sub-nanometer spatial resolution, which makes it possible to measure nanometer-scale particle and other three-dimensional (3D) samples. Most of these measurements only account for the size (width, length, etc.) of the sample in one direction or another, but rarely reconstruct the complete 3D shape of the sample. In a few cases, for example on simple integrated circuit structures, it is possible to extract 3D shape and size out of a single top-down image. It is even possible to do this with a few atoms worth of accuracy.

With complex objects, images taken from two or more directions or by slicing the sample either mechanically or by a focused ion beam and acquiring image stacks are necessary to reconstruct the 3D shape. Today the most developed electron microscopy 3D reconstruction methods use voxels, i.e., “volume pixels” for direct volume rendering using image stacks. These software packages can generate surface images, but do not work with SEM images that show only surfaces. Today there are a few software packages dedicated to 3D shape reconstruction using SEM images, but based on our results, these are not ready yet to work well with SEM images taken on nanometer-scale samples. We have investigated their performance with real images and with artificial images made by 3D Monte Carlo modeling. The simulated sample has a complex shape, and its images were simulated at tilt angles of +/- 5, +/- 10, +/- 20 and 0 degrees. We report on the results and possibilities of future 3D SEM measurements.

9636-32, Session 2

Fundamental comparison of electron imaging modes for dimensional metrology *(Invited Paper)*

Michael T. Postek, John S. Villarrubia, András E. Vladár, National Institute of Standards and Technology (United States); Atsushi Muto, Hitachi High Technologies America, Inc. (United States)

Achieving good SEM measurement accuracy depends on the quality of the acquired image influenced by vibration, drifts, sample contamination and charging, etc., and accounting for specimen-electron beam interactions. New acquisition methods and successful mitigation of detrimental effects can alleviate some of the imaging problems. But, another key element is the application of advanced electron beam-solid state interaction modeling, such as the NIST JMONSEL model to interpret and account for the physics of the signal generation, and help to understand and minimize the various contributions to measurement inaccuracy.

This work is a fundamental comparison of secondary (SE), backscattered (BSE) and low-loss (LLE) electron signals acquired on a new instrument that has high-angle BSE and energy-filtered LLE detectors. Early work indicated that the LLE signal could be advantageous for metrology. When that work was first done, it was very difficult to obtain the needed information because of poor signal-to-noise ratio (SNR) and other instrument-specific geometric limitations. LLE imaging is difficult because LLE represent a small and hence inherently noisier subset of all BSE that have undergone only minimal inelastic interactions with a sample and therefore carry high-resolution, surface-specific information. The use of the conventional backscattered electron signal was shown to be beneficial at low landing energies using a microchannel-plate electron detector. In that work, collection and comparison of the BSE generated images of line structures measured about 10 % narrower, compared to the width of measured SE images [9]. Due to the enhanced emission of low-energy (typically less than 10 eV) electrons at the sides and corners, there are common circumstances in which the SE intensity increases more abruptly at an edge than the BSE intensity. If width assignments are based on an intensity threshold, SE images would then be interpreted as showing a wider feature than the BSE image. It was anticipated that LLE signal would provide results similar to BSE results. A field emission scanning electron microscope, equipped with a high-angle and energy-filtered backscattered electron detector, was used to compare the SE, BSE and LLE signals for dimensional measurements of the NIST RM 8820 magnification calibration sample. The design of the new in-lens energy filtered detector improves the LLE signal-to-noise ratio and reduces the geometrical limitations of the early LLE detectors allowing for the first time, point-by point measurement data were able to be obtained simultaneously on a sample using these electron collection modes.

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9636-4, Session 3

Does your SEM really tell the truth? How would you know? Part 4: Charging and its mitigation *(Invited Paper)*

Michael T. Postek, András E. Vladár, National Institute of Standards and Technology (United States)

This is the fourth of a series of tutorial papers discussing various causes of measurement uncertainty in scanned particle beam instruments, and some of the solutions researched and developed at NIST and other research institutions. Scanned particle beam instruments especially the scanning electron microscope (SEM) have gone through tremendous evolution to become indispensable tools for many and diverse scientific and industrial applications. These improvements have significantly enhanced their performance and made them far easier to operate. But, ease of operation has also fostered operator complacency. In addition, the perceived user-friendliness has reduced the need for extensive operator training. Unfortunately, this has led to the concept that the SEM is just another expensive digital camera or another peripheral device connected to a computer and that all of the issues related to obtaining quality data have been solved. Hence, a person using these instruments may be lulled into thinking that all of the potential pitfalls have been fully eliminated and they believe everything they see on the micrograph is always correct. But, as described in this and the earlier presentations, this may not be the case. Care must always be taken when quantitative data are being recorded. The first paper in this series discussed some of the issues related to signal generation in the SEM, including instrument calibration, electron beam-sample interactions and the need for physics-based modelling to understand the actual image formation mechanisms to properly interpret SEM images. The second paper, discussed another major issue confronting the microscopist: specimen contamination and methods of contamination elimination. The third paper discussed mechanical vibration and stage drift and some useful solutions and this fourth contribution discusses some of the issues related to specimen "charging" and some possible solutions for its mitigation.

9636-5, Session 3

Three-dimensional characterization of Gd nanoparticles using STEM-in-SEM tomography in a dual-beam FIB-SEM *(Invited Paper)*

Brandon Van Leer, Cedric Bouchet-Marquis, Huikai Cheng, FEI Co. (United States)

Serial sectioning using the FIB and subsequent imaging of the same FIB-exposed surface by both FIB microscopy and scanning electron microscopy (SEM) in a DualBeam has proven especially useful to study the three-dimensional (3D) morphology of complex engineered materials systems [1]. The technique was first introduced as an automated process in 2004 and since then has established itself as one of the primary applications for FIB and DualBeams [2]. While state-of-the-art systems can produce datasets with a z-axis slice thickness of 3-5 nm, FIB nanotomography remains a destructive technique and is limited in resolution by the z-axis slice thickness. Electron tomography is another technique used to visualize 3D structures with transmission electron microscope in TEM or STEM modes. Using a thin sample focused on a region of interest, the electron beam passes through the specimen incrementally tilting around the center of the region of interest as images are acquired sequentially. The resulting images are reconstructed into a 3D volume using a variety of algorithms including weighted back projection (WBP) [3], or Serial Iterative Reconstruction Technique (SIRT)[4]. Low energy STEM in SEM is a routine analysis in SEMs and DualBeam FIB-SEM instrumentation for morphological characterization and ultra high-resolution imaging. With a DualBeam or SEM configured with a solid state silicon diode STEM detector and a stage with adequate tilt freedom, it is possible to acquire a sufficient number of images for 3D reconstruction using STEM tomography in SEMs and DualBeam instruments.

A thin section sample of gadolinium nanoparticles ranging in size up to 50 nm mounted on an aluminum substrate was prepared using in-situ lift-out (INLO) by FIB [5]. The sample was thinned using 30 kV Ga+ FIB to approximately 125 nm. Using an in-situ stage with 360 degree continuous tilt, the thin section was imaged every 1 degree with 30 keV SEM and the STEM detector through approximately 125 degrees of tilt. The data set was then reconstructed into a 3D rendering using FEI's tomography reconstruction software, Inspect3D Express, and visualized using FEI's Avizo image and data analysis software. The technique and results compared with conventional STEM tomography using a 200 keV FEI Titan Themis TEM will be discussed.

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9636-6, Session 3

A novel approach to TEM preparation with a (7-axis stage) triple-beam FIB-SEM system *(Invited Paper)*

Jamil J. Clarke, Hitachi High Technologies America, Inc. (United States)

Preparation of lamellae from bulk to grid for Cs-corrected Transmission Electron Microscope (TEM) observation has mostly become routine work on the latest FIB-SEM systems, with standardized techniques that often are left to automation for the initial steps. The finalization of a lamella however, has mostly become, non-routine, non-repeatable and often driven by user experience level in most cases to produce a high quality damage-less lamella. Materials processing of the latest technologies, with ever-shrinking nano-sized structures pose challenges to modern FIB-SEM systems. This can often lead to specialized techniques and hyper-specific functions for producing ultra-thin high quality lamella that often are lab specific, preventing practical use of such techniques across multiple materials and applications. Several factors that should be incorporated in processing fine structured materials successfully include how the use of electron and ion scan conditions can affect a lamella during ion milling, the type of ion species applied for material processing during the finalization of a lamella with gallium ions or of a smaller ion species type such as Ar/Xe, sample orientation of the lamella during the thinning process which is linked to ion beam incident angle as a direct relationship in the creation of waterfall effects or curtaining effects, and how software can be employed to aid in the reduction of these artifacts with reproducible results regardless of FIB-SEM experience for site-specific lift outs. A traditional TEM preparation was performed of a fine structure specimen in pursuit of a process technique to produce a high quality TEM lamella which would address all of the factors mentioned. These new capabilities have been refined and improved upon during the FIB-SEM design and development stages with an end result of a new approach that yields an improvement in quality by the reduction of common ion milling artifacts such as curtain effects, amorphous material, and better pin pointing of the area of interest while reducing overall processing time for the TEM sample preparation process and enhancing repeatability through ease of use via software controls. The development of these new technologies, incorporating a 3rd Ar/Xe ion beam column in conjunction with the electron and Ga+ ion beam column, a 7-axis stage for enhanced sample orientation with tilt functions in two axes and automated swing control along with a host of additional functions which address the factors aforementioned such as electron and ion scan techniques and curtain effect removal by the use of hardware and software components that are key to reduce typical FIB related artifacts,

called "ACE [Anti Curtaining Effect] Technology" are explained. The overall development of these technologies are to address a significant point that productivity, throughput and repeatability are comprised by synergy between the user, application, software and hardware within a FIB-SEM system. The latest Hitachi FIB-SEM platform offers these innovations for reliability, repeatability and high quality lamella preparation for Cs-corrected (S)TEMs.

9636-7, Session 3

Molecular dynamics study of crystalline orientation effect in scanning ion microscopes

Kaoru Ohya, The Univ. of Tokushima (Japan)

The scanning ion microscope (SIM) constructs an image of a sample surface by scanning an ion beam across the surface and detecting secondary electrons (SEs) and backscattered ions (BIs) from it, similarly to conventional scanning electron microscopes (SEMs). Channeling contrast is perhaps the most striking contrast mechanism in the SIM and derives from the variation in crystallographic orientation of crystalline samples relative to the beam. In this paper, we presents a simulation study of crystalline orientation effect for ion beams of He, Ne, and Ga at tens-of-keV energies relevant to the SIM.

In order to calculate the BI yield, ion trajectories were pursued by solving kinetic equation of motion in a small cell of W with periodic boundaries conditions of the sides of the cell [1]. The temperature control of the cell used a Langevin thermostat [2], dissipating the excess heat between impacts. The forces were calculated from empirical bond-order potentials for W-W system [3] and the Ziegler-Biersack-Littmark potentials [4] for He-W, Ne-W, and Ga-W systems. The ions were assumed to lose energy continuously in the trajectory from electronic interaction of Lindhard-Scharff type [5]. The SE yield was simply calculated through a semi-empirical scheme, assuming that (1) the number of SEs produced along the ion trajectory is proportional to the electronic energy loss, (2) the SEs decay exponentially toward the top surface of the cell, and (3) the transmission probability through the surface is 0.5. The important quantities are the average energy required to produce one SE and the decay length of the SE, which were taken to be 78 eV and 0.75 nm, respectively, for He impact on W, according to ref. [6].

Oscillatory trajectories were clearly observed for the ions penetrating along a crystallographic axis of W. Most of He ions were directed by the atomic row and the large angle scattering from the atoms became small. However, because of the increase of the scattering with increasing mass of the ions, the near-surface trajectories laterally distributed more for Ga ions than for He ions. Both BI and SE yields showed a general increase with the angle of incidence, while showing a sharp reduction of the yields when the incidence was directed along the atomic row. The reduction was clearer in the BI yield of heavy ions whereas it was not clearly found in the SE yield for light lions. Furthermore, the thermal and recoiling motions of the cell atoms caused the reduction to loss sharpness.

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9636-8, Session 4

Correlative microscopy including CLSM and SEM to improve high-speed, high-resolution laser-engraved print and embossing forms

Markus Bohrer, Dr. Bohrer Lasertec GmbH (Austria); Michael Schweitzer, Carl Zeiss SMT GmbH (Austria); Robert Nirnberger, Carl Zeiss GmbH (Austria); Bernhard Weinberger, Dr. Bohrer Lasertec GmbH (Austria)

The industrial market for processing large-scale films has seen dramatic changes since the 1980s and has almost completely been replaced by lasers and digital processes. A commonly used technology for engraving screens, print and embossing forms in the printing industry, well known since then, is the use of RF-excited CO2 lasers with a beam power up to about 1kW, modulated in accordance to the pattern to be engraved. Future needs for high-security printing (banknotes, security papers, passports, etc.) will require laser engraving of at least half a million or even more structure elements with a depth of some µm and up to 500 µm. Industry now wants photorealistic pictures in packaging design, which requires a similar performance. To ensure "trusted pulses" from the digital process to the print result the use of correlative microscopy (CLSM and SEM) is demonstrated as a complete chain for a correlative print process in this paper.

9636-9, Session 4

Sub-diffraction-limit imaging using mode multiplexing

Nan Wang, Jinping He, Jun Miyazaki, The Univ. of Electro-Communications (Japan); Hiromichi Tsurui, Juntendo Univ. (Japan); Takayoshi Kobayashi, The Univ. of Electro-Communications (Japan)

To investigate nanoscale interaction or colocalization in biological tissues and microstructure materials, multi-color superresolution far field optical microscopy could be a powerful tool. However the microscopes and fluorophores staining in the sample presently used to approach this goal still have some limitations. Firstly, optical superresolution microscopies, like STED, SIM, PALM, STORM and others, which have achieved great success in break the diffraction-limit, are expensive and inflexible to build and maintain. Especially when they are applied for multi-color imaging, the schemes and their implementations are quite costly and complicated. Secondly, organic dyes, which have been widely used as fluorescent labels in multi-color imaging for decades, introduce inevitable fluorescence crosstalk due to their broadband emission spectra and are easily photobleached. In comparison, quantum dots (Qdots), which have narrowband fluorescence spectra that can be easily separated by mirrors, broadband absorption spectra that can be possibly excited by only single laser wavelength and are highly photochemistry stable, have not been used in multi-color superresolution microscopes, like STED, PALM or STORM, due to their special requirements of probes. Meanwhile, multi-color imaging with SOFI or SIM with Qdots has not been reported yet as far as we know. In comparison, subtraction microscope can provide a simple approach to obtain multi-color superresolution imaging in combination with generalized fluorescent probes.

The principle of the subtraction microscope can be described as: the sample is excited with the same laser wavelength but with two different focal spot shapes, which are usually a Gaussian distribution and a donut intensity distribution. The obtained fluorescence image by the latter is subtracted from that by the former. Then the produced image has a higher resolution compared with the conventional fluorescence image.

Until now, one color subtraction microscopes acquiring the images of the two excitation spots frame-by-frame or line-by-line has been reported. Nevertheless, a repeating scan creates uncertainties due to mechanical drift or changes in the properties of the fluorescent probes, even in unrealistically fast imaging. Especially for moving targets or live cell imaging, the ability to simultaneously acquire and process the images excited by the two beams is essential. We have demonstrate pixel-by-pixel processed subtraction microscopy by multiplexing the excitation laser spots with Gaussian and donut shapes and then demultiplexing the fluorescent signals using lock-in amplifiers. Using a homemade microscope, fluorescent polystyrene beads and a slice of brain tissue of a mouse are imaged with sub-diffraction-limit resolution. A "moving" sample is also imaged to inspect the robustness of the microscope. The corresponding spatial resolution improvement is about 26% when the same excitation power of two beams is implemented. The time resolution

of the scan can also be improved by a factor of 2 resulting from the simultaneous measurement.

With upgrade of the microscope, two-color imaging of fluorescent beads stained with organic dyes and mesenteric lymph nodes of a mouse labeled with Qdots have also been achieved. Resolution enhancement of 20% ~ 30% in both of the two color channels is obtained compared with confocal microscopes. Theoretical investigation about the subtraction threshold is also discussed.

9636-10, Session 4

Investigation of electron beam irradiation effect on pore formation

Seong Soo Choi, Myoung Jin Park, Chul Hee Han, Sun Moon Univ. (Korea, Republic of); Sung In Kim, Jung Ho Yoo, Kyung Jin Park, National Nanofab Ctr. (Korea, Republic of); Nam Kyou Park, Seoul National Univ. (Korea, Republic of); Yong-Sang Kim, SungKyunKwan University (Korea, Republic of)

We investigated the influence of electron beam irradiation on Au nanopore formation. The local temperature rise would depend upon dwell time of scanning beam and temperature diffusivity of the specimen. For the temperature rise above Au melting points, high enough to make Au viscous, Au atom would migrate into the pore, or evaporate under high vaccum environments. Temperature equilibrium time in the nanometer thick membrane specimen is order of ~ nanosec, too fast to consider scanning speed or dwell time. However, we found that resizing of the FIB drilled aperture depends upon distance between the aperture and scanning area, and resizing of the aperture for 7 micrometer away from the aperture was not observed. We find that the diffused membrane contains Au clusters and Au particles on carbon membrane due to Ostwald ripening after the sample was kept in a desiccator for long period of time.

9636-11, Session 4

Investigation of all-optical micro- and nanolithography by electric field intensity gradient

Ugis Gertners, Zanda Gertnere, Janis Teteris, Univ. of Latvia (Latvia)

All-optical or direct writing technique is a comparatively new solution for lithography and provides new experimental techniques for better understanding of the interaction between the light and matter. The demand of lower cost surface-relief based optical instruments such as grating-based resonators or filters for waveguides, diffractometers, spectrometers, etc. is one of the main driving forces for the investigation of direct light-induced relief formation. The most common techniques for fabricating and investigating these surface-relief gratings involve an interferometric or holographic recording setup. We have investigated that the light-induced mass transfer process strongly depends on the material itself and polarization of the light. The behavior of mass transfer and thus the resulting recording could be related to interaction between the polar photo-induced defects and the polarized electric field of recording beam. It has been shown that the mass transfer can be directed both ways – towards or away from the electric field intensity gradient. The evolution of surface relief in dependence from the recording time and polarization has been investigated in detail. The mechanism of the direct recording of surface relief on amorphous organic and inorganic films based on the photo-induced plasticity has been discussed.

9636-12, Session 5

Very-high energy (300-400 keV) SEM imaging of Cu interconnects *(Invited Paper)*

Lynne Gignac, Christopher M. Breslin, Jemima Gonsalves, Franco Stellari, Chung-Ching Lin, IBM Thomas J. Watson Research Ctr. (United States)

Most commercial scanning electron microscopes (SEM) have a maximum incident electron beam energy of 30 keV. To perform higher energy SEM analysis, secondary electron (SE) and backscattered electron (BSE) detectors can be installed in a transmission electron microscope (TEM)/scanning TEM (STEM) and SEM imaging can be performed using incident beam energies between 100-400 keV. The benefits of high energy SEM include atomic resolution SE imaging(1) and imaging of structures several microns below the surface by detecting BSE(2). In this paper, sub-surface imaging of multilayered structures is further studied in TEM/STEM systems with incident beam energies between 300-400 keV by analyzing a fully built, 90 nm technology Si chip with 9 interconnect levels (8 um thick) using SE, BSE and STEM bright field (BF)/dark field (DF) signals with the chip oriented with top side and then bottom side up relative to the incident electron beam. To obtain useful images from the backside of the chip, the chips were first mechanically thinned and then etched using XeF2 gas. The high energy SE images showed surface details and near surface interconnect structures. The images collected with a semiconductor BSE detector showed no surface details but interconnect structures >5 um below the surface could be detected. Since the sample was 8 um thick, the incident electron beam was highly scattered through the sample thickness and the BF and DF images had equivalent contrast for most standard camera lengths used for thin samples. When the camera length was decreased, the DF image had the expected inverted contrast from the BF image but the resulting image quality was poor with low signal to noise.

Focused ion beam (FIB) SEM tomography was performed on a region of the chip that was first fully characterized by 300-400 keV SE, BSE, STEM and TEM imaging. The 3-dimensional reconstruction from the SEM image series was used to determine the depth of the sub-surface features. By BSE or BF STEM, features near the top surface were imaged but sub-surface features could be seen with diminishing resolution with depth due to beam scattering. By TEM, features on the bottom surface of the sample could be seen. When comparing BF STEM images taken with top side up and TEM images with the bottom side up, similar features were seen in the images but the TEM images had reduced resolution due to chromatic aberrations from projector lenses.

Very large field of view (300 x 300 um) BSE images were collected using STEM low magnification mode (objective lens off and objective mini lens excited) with the added benefits of an extremely parallel beam (14 uR convergence angle) that allowed large features in the full 8um interconnect stack to be in focus. In this mode, the BSE image was identical if the top or bottom sides of the sample were up in the microscope.

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9636-13, Session 5

Application of gas field ion source to photomask repairs *(Invited Paper)*

Fumio Aramaki, Tomokazu Kozakai, Osamu Matsuda, Hiroshi

Oba, Yasuhiko Sugiyama, Kazuo Aita, Anto Yasaka, Hitachi High-Tech Science Corp. (Japan)

Focused ion beam (FIB) systems are well known as tools for high resolution imaging and processing. Typical FIB systems use gallium ions (Ga+) emitted from a liquid metal ion source (LMIS), but recently some ones have used helium ions (He+) emitted from a gas field ion source (GFIS). We have developed a novel GFIS system that can emit not only He+ but also hydrogen ions (H2+) and nitrogen ions (N2+). H2+ has lower irradiation damage than He+, because H2+ beam is lighter than He+. N2+ takes sputtering effect, but He+ does not take it.

Meanwhile, scanning microscopes have been used for repairing pattern errors on photomasks for semiconductor manufacturing. Specifically, the repair systems etch superfluous film and deposit film at missing area. Those beams have become to more precise depending on the shrinkage of device design rules. At present, most of defects on high-end masks are repaired with EB systems, but there are two issues. Firstly, the ratio of the etching rate of some advanced films to that of quartz substrate is less than 2, but it is not high enough to prevent over/under-etching. Secondly, the minimum etching dimension is approximately between 20 and 30 nm, but it is not small enough to repair the next generation masks.

We have solved the above two issues by using N2+ beam emitted from GFIS. The ratio of the etching rate with N2+ beam of the above films to that of quartz was more than 4. The minimum etching dimension of N2+ beam was less than 10 nm. Furthermore, we have verified the feasibility of GFIS technology for repairing extreme-ultraviolet (EUV) lithography masks that are the most promising candidate of the next generation masks. It was found that He+ beam is not applicable to EUV masks owing to serious irradiation damage on reflection layers, but H2+ beam showed good repair results without significant damage.

The above results demonstrate that GFIS technology is a promising candidate of repairing the next generation masks.

9636-14, Session 6

A compilation of cold cases using scanning electron microscopy at the University of Rhode Island

Michael J. Platek, Otto J. Gregory, The Univ. of Rhode Island (United States)

This presentation will address how the Scanning Electron Microscope was used to evaluate evidence for two cold cases. It will discuss the methodology used to evaluate the evidence and discuss the findings of the case. It will highlight the importance of using the electron microscope as a tool in helping to solve these cases.

9636-15, Session 6

SEM/EDS analysis for problem solving in the food industry

Wayne D. Niemeyer, McCrone Associates, Inc. (United States)

Scanning electron microscopy (SEM) in conjunction with energy dispersive x-ray spectrometry (EDS) is a powerful, often non-destructive, instrumental analysis tool to provide information about:

• Identification of inorganic (and some organic) materials found as foreign contaminants in food products returned by consumers or detected during quality control inspections in the production facilities

• Identification of wear particles found in production lines

• Distribution of materials within a matrix

• Particle sizing with or without “chemical typing”

• Corrosion and failure analysis of production equipment

The identification of materials by SEM/EDS is accomplished through a combination of morphology by SEM imaging and the elemental composition of the material by EDS. Typically, the EDS analysis provides a qualitative spectrum showing the elements present in the sample. Further analysis can be done to quantify the detected elements in order to further refine the material identification. Metal alloys can often be differentiated as in an example of determining if a metal is a 316 alloy stainless steel or 304 alloy stainless steel. The 304 stainless steel contains less nickel and no molybdenum compared to the 316 stainless steel. Glass types can be identified by the elemental composition where the detected elements are quantified as the oxides of each element. In this way common window glass is distinguishable from insulation glass used in many ovens. A material can be identified as paint by the composition and particle size of inorganic fillers/pigments/extenders.

Wear particles or fragments from breakage can find their way into food products. SEM/EDS analysis of the materials is an important resource to utilize when trying to determine the original source. Suspected source materials can then be sampled for comparative analysis.

EDS x-ray mapping is another tool that is available to provide information about the distribution of materials within a matrix. For example, the distribution of inorganic ingredients in a dried cereal helps to provide information about the grind and blend of the materials.

SEM/EDS particle sizing of ingredient powders provides information about the size distribution, but also the EDS analysis can be used in combination with the SEM particle sizing to provide the elemental composition information of individual particles. This can be extremely useful in determining the purity of the ingredient powder.

9636-16, Session 6

Observations on the variability in deposition and recovery of gunshot residue particles for fabric targets within 10 meters of a discharged firearm

Andrew Wolf, Ctr. of Forensic Sciences (Canada); Victoria Del Plavignano, Univ. of Ontario Institute of Technology (Canada)

Gunshot residue (GSR) produced by the cartridge primer during the discharge of a firearm is commonly used as an indicator of presence at a shooting or contact with an object previously exposed to GSR. Samples from the hands and clothing of someone suspected of discharging a firearm are commonly collected as part of police investigations. The purpose of this study was to evaluate the spatial variation in GSR particle deposition and to quantify the collection efficiency of GSR particles from various fabrics using tape-lift sampling.

GSR was produced in a firing range (Centre of Forensic Sciences) by discharging ‘American Eagle REM .45 auto’ ammunition using a ‘Para Ordnance P12-45’ semi-automatic handgun. GSR was collected directly on 25mm diameter SEM stubs bearing 3M’s 415 double-sided adhesive tape. These stubs were also used to collect GSR from swatches (15 x 15 cm) of six different fabrics (synthetic leather, nylon, denim, cotton, plush and fleece). The stubs and fabrics were mounted on boards approximately 75 x 100 cm in size. Each experiment consisted of discharging at least 10 rounds of ammunition and waiting at least 10 minutes before removing samples from the range. Particles containing Pb, Ba and Sb were counted as characteristic GSR particles. These were identified using an ASPEX scanning electron microscope equipped with automated GSR search software, a thin-window energy dispersive X-ray detector and a back-scatter electron detector.

In an experiment to observe the effect of air-flow on GSR deposition, stubs were mounted on a vertical board located ~1.5m to the right of the firearm. When ventilation was turned off, at least 4 GSR particles were identified on each stub. With ventilation at a ‘normal flow rate,’ GSR was not identified on stubs in the same positions. Ventilation remained off for subsequent experiments.

In an experiment to observe the effect of substrate orientation, stubs were mounted on horizontal and vertical boards located ~10m downrange from the firearm. At least 15 GSR particles were identified on stubs mounted on the horizontal board, yet no GSR particles were identified on stubs mounted on the adjacent vertical board.

Another experiment was performed with an array of 24 fabric swatches

pinned to a horizontal board, at a height of 0.7m, positioned ~2.5m downrange from the firearm. A stub was positioned in the centre of each swatch to serve as a direct deposition reference. Collection efficiency for each swatch was determined by dividing the amount of GSR identified on tape-lift samples from the fabric by the amount identified on the reference stub.

The number of GSR particles identified on the reference stubs varied from 1 to 54. For pairs of single dab collections at a separation of ~6cm, one-third of the fabric samples had adjacent zero and non-zero GSR counts. The amount of GSR identified on stubs undergoing 10-dab collections varied as much as that observed for single dab samples, but GSR was identified on all of the 10-dab samples. While the results overlapped for all of the fabrics, average collection efficiencies were 55% for nylon, 50% for synthetic leather and ranged from 24% to 35% for denim, cotton, plush and fleece. Greater recovery from nylon and synthetic leather can be attributed to more particles remaining on the outer surface of the fabric and a higher degree of contact between the fabric and the tape-lift sampler. The results demonstrate an inherent uncertainty in the amount of GSR deposited on objects in the vicinity of firearm discharge and in the amount recovered from clothing.

9636-17, Session 6

Exploration of mXRF analysis of gunshot residue from cartridge cases

Martin Janssen, Alwin Knijnenberg, Amalia Stamouli, Netherlands Forensic Institute (Netherlands)

The investigation of garment and human tissue originating from a victim of a shooting incident can provide crucial information for the reconstruction of such an incident. Determining the entries and exits of bullet trajectories, the shooting distances and the elemental composition of ammunition are prime examples of such information. Previous work [1-6] on 2D-mXRF shows promising results in avoiding the biggest drawbacks that accompany the chemographic methods and SEM/EDX methods that are currently in use in forensic laboratories worldwide.

As 2D-mXRF is new in the forensic field, the Netherlands Forensic Institute (NFI) is exploring [7] this technique and its implementation in case investigations. In our previous work [7], we explored the use of 2D-mXRF as a fast screening tool for the elemental composition of GSR originating from cartridge cases. During these first experiences, the main problem that was encountered was related to the contribution of the sampling materials to the XRF spectra. In the current work we will present the next steps taken in applying the 2D-mXRF technique as a screening tool.

In addition to the application as a screening tool, we started exploring the possibilities of 2D-mXRF for the investigation of bullet holes mainly in construction materials. Recently, the Gunshot Residue Group of the NFI was asked to investigate several cases with potential bullet holes in walls and flooring. As such objects are often difficult to remove from crime scenes, often castings are made to enable investigations at the forensic laboratory. Furthermore these walls and flooring often have irregular surfaces, making them difficult for sampling with foils or even stubs and less suitable for direct application chemographic methods in which the material needs to be subjected to pressure in a clamping system. In this paper we present our first experiences with the investigations of castings using 2D-mXRF.

9636-18, Session 6

The use of commercial reference materials including the NIST Reference Manual (RM) 8820 SEM Artifact for SEM Calibrations in Forensic Laboratories *(Invited Paper)*

S. Frank Platek, U.S. Food and Drug Administration (United States); Michael T. Postek, András E. Vladár, National Institute of Standards and Technology (United States); Stefanie L. Heckman, U.S. Food and Drug Administration (United States)

Scanning electron microscopes (SEM) are workhorse instruments in many forensic laboratories. They are used for imaging and characterization of a wide variety of case-related samples. The data obtained are often used for judiciary purposes and hence, must be the highest quality possible. As with most scientific instruments, care must be taken in the operation and the calibration must be checked periodically. This was clearly defined in 2006, when the United States Congress mandated that the National Academy of Sciences (NAS) form a committee to examine and assess the resources, needs, and practices of the forensic science community. In 2009, as part of the extensive report "Overview of the NAS Report on Forensic Science in the U.S.", one line item noted a "Paucity of scientific research on validity and reliability of forensic disciplines and quantifiable measures of uncertainty".1 In light of the NAS report, one of the many issues considered has been an examination of scientific instruments and the accuracy of measurements produced by those instruments. The topic of this paper is to review selected standards used to evaluate the spatial measurements in the SEM. With the exception of a few studies to document on-screen measurement and magnification accuracy of commercially available SEMs, (Postek et al 2, 3), SEM users have often trusted the default instrument measurements as reliable. The Postek et al. studies demonstrated the variability in magnification accuracy possible for a number of SEMs, which therefore raises concerns about the reliability of measurements by the instruments.

For many SEM users, including most forensic laboratories, the accuracy of an instrument's on-screen measurements are currently verified using commercially available standards which may have varying levels of accuracy and traceability (e.g. non-certified, certified, National Institute for Standards and Technology - (NIST) traceable, non-traceable). Some ISO 17025-based accreditation organizations for forensic laboratories recommend or require the use of certified standards which are NIST-traceable. In some cases, accreditation organizations further require the SEM itself to be calibrated by an accredited calibration service before measurements can be documented in forensic case reports. This paper will explore the use of several commercially-obtained calibration standards, including the NIST RM 8820 SEM magnification calibration artifact4, and their applications in forensic laboratories.

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9636-19, Session 7

There's an electron microscope on that bus?! *(Invited Paper)*

Sarah Weisberg, Cell Motion Labs. (United States); Ben Dubin-Thaler, Cell Motion Labs. (United States); Robert Gordon, Hitachi High Technologies America, Inc. (United States)

In New York City, home to elite scientific research and cultural institutions, less than one third of students show proficiency on national science exams. Even our top-performing students are not competing successfully in national science competitions. The BioBus inspires the next generation of STEM leaders by clearing road blocks such as lack of equipment and lack of research experiences and getting students hands-on with an Hitachi TM3030 electron microscope while they work side-by-side with scientists in an authentic research environment. Every year,

more than 25,000 students board the solar-powered BioBus lab, where students use the Hitachi TM3030 to a) view the bacteria living on the shell of the crustaceans they were just viewing on light microscopes or b) view next generation nanofabricated particles that spark their imagination of what their future might hold as a materials scientist.

Hundreds of our most excited BioBus students take the next step towards a career in science at the BioBase, our brand new community science lab. At the BioBase, students work after school, on the weekends, and at summer camps on science projects such as a) using the Hitachi SEM to survey pollen grains from local community gardens and identify pollinator behaviors in collaboration with entomologists from Cornell University, b) characterizing nanoparticles fabricated by our academic partners at New York University Materials Research Science and Engineering Center, c) making new discoveries in C. elegans morphology in partnership with partners at Albert Einstein School of Medicine and d) learning how to preserve and sputter coat their own samples in collaboration with partners at Brooklyn College and Columbia University. Research at Columbia University's Teachers College has shown significant, positive impacts of the BioBus on students' science attitudes and behaviors in the short and medium term, and our students have gone on to perform research at and enroll in college at top-tier institutions including Columbia and Harvard. The BioBus serves as a model of how industry and academia can partner with a community non-profit to work within the existing school system and create dynamic, transformative learning environments that inspire the next generation of scientists.

9636-20, Session 7

Hitachi TM3030 engages at the nexus of cross-curriculum teaching and vertical articulation *(Invited Paper)*

Dave E. Menshew, James C. Enochs High School and Brandman Univ. (United States)

In this investigation, the researcher will show how the Hitachi TM3030 scanning electron microscope formed a nucleus to engage students in science, technology, engineering and mathematics learning in grades 5 to university.

Learners served include at risk, serving consequences for behavioral issues, gifted and talented as well as resource and mainstream students including seniors engaged in capstone research projects and university learners who found the device relevant to their current learning but unavailable on their home campus.

The device also played a central role in the development of a model cross-curricular unit engaging students in such diverse classes as agriculture, art, biology, drama, dance, language arts and mathematics. Vertical articulation included elementary schools, high school and multiple universities. The work will be included as an exemplar in a university teacher-training program.

Metrics show that the device raised student awareness of STEM career options, provided enhance engagement in both investigation and technology, and proved to be a highly positive, student-directed experience.

9636-21, Session 7

Using a university characterization facility to educate the public about microscopes: light microscopes to SEM *(Invited Paper)*

Nancy Healy, Walter Henderson, Georgia Institute of Technology (United States)

The National Nanotechnology Infrastructure Network (NNIN) is an integrated partnership of 14 universities across the US funded by NSF to support nanoscale researchers. The NNIN education office is located at the Institute of Electronics and Nanotechnology at the Georgia Institute of Technology. At Georgia Tech we offer programs that integrate the facility and its resources to educate the public about nanotechnology. One event that has proved highly successful involves using microscopes in our characterization suite to educate a diverse audience about a variety of imaging instruments. As part of the annual Atlanta Science Festival we provided an event "What's all the Buzz about Nanotechnology?" which was open to the public and advertised through a variety of methods. During the event, we provided hands-on demos, cleanroom tours, and activities with three of our microscopes in our recently opened Characterization Facility: 1. Keyence VHX-600 Digital Microscope; 2. Hitachi S-4700 FE-SEM; and 3. Hitachi TM 3000. During the two hour event we had approximately 150 visitors including many families with school-aged children. Visitors were invited to bring a sample for scanning with the TM-3000. This presentation will discuss how to do such an event, lessons learned, and visitor survey results.

9636-22, Session 7

How a lesson on microscopes supports learning about light in elementary schools *(Invited Paper)*

Joyce Allen, Nancy Healy, Georgia Institute of Technology (United States)

The National Nanotechnology Infrastructure Network (NNIN) is an integrated partnership of 14 universities across the US funded by NSF to support nanoscale researchers. The NNIN education office is located at the Institute of Electronics and Nanotechnology at the Georgia Institute of Technology. As part of our programs at Georgia Tech, we provide lessons and activities for the K-12 community which are presented at workshops and school programs. Two of our learning goals when working with elementary students is that they understand that amazing things happen that are too small to be seen with just their eyes and that scientist can use tools to observe small things. One lesson we have developed and tested is Taking a Closer Look at Objects written for the elementary grades. Our purpose for creating this lesson was to help students understand that they often need to use tools to help them understand the world around them. Some of these tools will help them see parts of the world that they would otherwise miss which we hope will lay the foundation for understanding nanoscale objects as they move into higher grades. These tools include magnifying glasses and microscopes. This presentation will discuss how this lesson provides students an opportunity to learn how microscopes and lenses make small objects in our world visible. We will show how the lesson can be conducted as a learning center or as a teacher-led whole class instruction. Results of our testing of the lesson with elementary students will be presented.

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9636-23, Session 8

Continuous monitoring of tip radius during atomic force microscopy imaging *(Invited Paper)*

Jordi Fraxedas, Institut Català de Nanociència i Nanotecnologia (ICN2) (Spain); Francesc Pérez-Murano, Ctr. Nacional de Microelectrónica (Spain); Federico Gramazio, Institut Català de Nanociència i Nanotecnologia (ICN2) (Spain); Matteo Lorenzoni, Ctr. Nacional de Microelectrónica (Spain); Enrique Rull Trinidad, Urs Staufer, Technische Univ. Delft (Netherlands)

Tip wear is intimately related to the operation of an atomic force microscope (AFM). Initially sharp tips become modified by the contact with the surfaces under study thus changing irreversibly their shape and condition with time. Tip-induced effects, including both the damaging of the tip and of the explored surface, are concerns as old as AFM itself, so that several strategies have been developed in order to monitor the tip condition or protect it by making it more robust. Amplitude modulation (AM)-AFM was implemented with the aim to strongly reduce tip wear, eliminating the shear stresses acting on the tip due to sliding,

through the intermittent contact. However, such reduction is only partial because in practice tip wear is still a problem even in the gentle AM-AFM mode. Here, we present a continuous tip monitoring method based on the use of higher harmonics, which are generated in the repulsive regime as a result of the nonlinear interactions between the tip and the studied surface. The underlying principle is the monotonically increase of the amplitudes of the higher harmonics for increasing values of the tip radius. Due to the involved low amplitudes this effect can be more efficiently observed when higher harmonics are in resonance with flexural eigenmodes of the cantilever. We have applied this method to commercial rectangular microfabricated silicon cantilevers in the 3-50 N/m range (50-400 kHz) with ultrasharp tips (tip radius below 10 nm) focusing in the resonance of the 2nd flexural mode and the 6th harmonic using both polymer and silicon surfaces, thus covering the 1-130 GPa Young's modulus range. We have observed both experimentally and by means of simulations using the Virtual Environment for Dynamic AFM (VEDA) open code that the amplitude of the 6th harmonic increases for increasing tip radius and that this increase becomes more obvious for smaller tip radii (below 20 nm). This allows the simultaneous acquisition of topographic and higher harmonic images thus continuously controlling the state of the tip.

9636-24, Session 8

Correlated nanoparticle height and width measurements in atomic force microscopy *(Invited Paper)*

Natalia Farkas, John A. Dagata, National Institute of Standards and Technology (United States)

The measurement approach typically prescribed for sizing spherical nanoparticles by atomic force microscopy (AFM) is based on a single value of the height obtained from a three-dimensional image of the particle [1,2]. The consequence is that almost all information about the particle is left unused. While tip convolution is understandably a concern, it is not an insurmountable problem, at least in the case of the most important (nearly) spherical particle reference standards. In this talk, we present a complementary measurement model based on correlated height and width measurements of individual, discrete particles. First, modeling of three significant contributions to measurement uncertainty is reviewed. Previous work with polystyrene (PSL) reference materials established [3]: 1) The validity of a two-parameter AFM probe tip model and operational limits for AFM probes due to fracture and wear using an Si/SiO2 multilayer tip characterizer; 2) A means of detecting and minimizing particle-tip deformation induced by dynamic AFM imaging of the particle; and, 3) An estimate for particle-substrate deformation by adhesive forces acting between the particle and substrate during sample preparation. These estimates of probe and deformation influence parameters are then incorporated into the analysis of correlated AFM measurement data. Finally, the capability of this procedure to serve as a statistical error-correction function for AFM particle size metrology is demonstrated for a series of well-characterized PSL reference materials (diameter range of 30 nm to 100 nm) and obtained using a number of individual AFM probe tips.

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9636-25, Session 8

Lateral tip control effects in CD-AFM metrology: The large tip limit *(Invited Paper)*

Ronald G. Dixson, National Institute of Standards and Technology (United States)

Critical dimension atomic force microscopes (CD-AFMs) use flared tips and two-dimensional sensing and control of the tip-sample interaction to enable scanning of features with near-vertical or even reentrant sidewalls. Features of this sort are commonly encountered in semiconductor manufacturing and other nanotechnology industries. The National Institute of Standards and Technology (NIST) has experience in the calibration and characterization of CD-AFM instruments and in the development of uncertainty budgets for typical measurands in semiconductor manufacturing metrology.

Sidewall sensing in CD-AFM usually involves lateral dither of the tip, which was the case in the first two generations of instruments. Current, third generation instruments also utilize a control algorithm and fast response piezo actuator to position the tip in a manner that resembles touch-triggering of coordinate measuring machine (CMM) probes. All methods of tip position control, however, induce an effective tip width that is larger than the actual geometrical tip width.

NIST has been investigating the dependence of effective tip width on the dither settings and lateral stiffness of the tip as well as the possibility of material effects due to sample composition. Generally, we have concluded that these effects will not generally result in a residual bias, provided that the tip calibration and sample measurement are performed under the same conditions.

To further validate our prior conclusions about the dependence of effective tip width on lateral stiffness, we recently performed experiments using a very large non-CD tip with an etched plateau of approximately 1.8 µm width. The lateral stiffness of these tips is nearly three orders of magnitude larger than typical CD-AFM tips – and the results supported our prior conclusions about the expected behavior for larger tips.

9636-26, Session 9

SPM metrological assurance using a heterodyne interferometer

Tatiana Kazieva, NRNU MEPhI (Russian Federation); Andrey P. Kuznetsov, Konstantin L. Gubskiy, Vladimir N. Reshetov, National Research Nuclear Univ. MEPhI (Russian Federation); Maria Ponarina, NRNU MEPhI (Russian Federation); Andrey Antonov, LaserEye LLC (Russian Federation); Alexey Useinov, TISNUM (Russian Federation)

We present the developed three-coordinate laser heterodyne interferometer for nanometer-scale displacement measurement traceability to the primary standard of meter for use in the scanning probe microscopes, scanning electron microscopes, and coordinate measuring machines.

Distinctive features of the interferometer are the versatility, compactness, high resolution and high accuracy.

Light source is a frequency-stabilized 1-mW-power He-Ne laser with the wavelength of 632.991084 nm (relative instability of frequency for 8 hours is 10?9). Thus the interferometer provides traceability of measurements in the nanometer and micrometer range to the primary standard of meter.

Signal processing system is based on the quadrature direct analog-to-digital conversion of the phase-modulated signals, followed by the digital processing of the informative samples. Correlation principle of detection provides high performance and the record-breaking phase measurement accuracy (up to 10-5 radians) with significantly reduced signal-to-noise ratio in the photocurrent receiver.

Triple prisms are used as reflectors, measuring the displacement of the interferometer. They are rigidly fixed on the platform, above the piezoelectric table. Two of them are mounted on the adjacent side faces

of the platform, and the third one - orthogonally to the former in the bottom of the platform, in the piezoelectric section. The mirror prisms are situated above it. The platform is placed on the piezoelectric table so that the axis of symmetry of the triple prisms extends through the center of rotation of the piezoelectric section. This arrangement minimizes the Abbe error encountered when using other optical schemes.

Thanks to elaborately designed optical unit and novel detection system, our device guarantees high measurement accuracy (~ 0.1 nm) and high resolution (0.01 nm).

The study of the interferometer's metrological characteristics was performed on the probe microscope NanoScan-3D with integrated interferometer using three reference structures (gratings). The results were compared with the measurements of the same gratings samples made at Physikalisch-Technische Bundesanstalt (PTB) (Germany) using a metrological probe microscope. For all three gratings, the values obtained from NanoScan-3Di fall into PTB calibration interval within 95% confidence.

Metrological probe microscope equipped with the three-coordinate laser interferometer belongs to Russian national primary special standard unit of length in the measurement of roughness parameters in the range of 1–1000 nm (VNIIMS, Russia).

Three coordinate laser heterodyne interferometer belongs to Russian national primary special standard unit of hardness for Martens scales and scale of nanoindentation (VNIIFTRI, Russia).

9636-28, Session 9

Initial results of photomask linewidth comparison by PTB and NIST *(Invited Paper)*

Detlef Bergmann, Bernd Bodermann, Harald Bosse, Egbert Buhr, Gaoliang Dai, Physikalisch-Technische Bundesanstalt (Germany); Ronald G. Dixson, National Institute of Standards and Technology (United States); Wolfgang Hässler-Grohne, Kai Hahm, Matthias Wurm, Physikalisch-Technische Bundesanstalt (Germany)

Precise dimensional control is crucial in photomask manufacturing and the comparability of results of different measurement methods, [1] the possibility of a reference measurement method [2] and of combined or multi-tool metrology solutions, [3] i.e., using the results of different methods have been investigated in recent years. PTB and NIST are undertaking a bilateral comparison of dimensional metrology performed at both institutes on a chrome-on-glass photomask.

The mask used for this comparison is a PTB design [4] that was manufactured by the Advanced Mask Technology Center (AMTC) in Dresden, Germany. We have compared the results down to the nominal 100 nm line features on this mask (isolated and dense, clear and opaque structures). The linewidth or critical dimension (CD) measurements on the photomask standard were performed using multiple techniques – including mainly CD-AFM, CD-SEM and transmission optical microscope characterizations. The definitions of the measurands were stated, as e.g. width of the line at 50 % of the line feature height.

For every measurement method applied, a suitable model for the determination of the measurand from the microscope image has been applied. For each of the linewidth measurement results the associated measurement uncertainty values were evaluated according to accepted international guidelines [5].

The results of the first iteration indicate good agreement, within the uncertainties, for measurands such as middle width. But for measurands such as sidewall angle, the results suggest that both institutes need to further evaluate the measurand definition and it's applicability to their data and uncertainty models. This paper presents and discusses the comparison of measurement results with their associated measurement uncertainty values.

Looking ahead, this bilateral photomask comparison between PTB and NIST also serves as a preparation for a future international comparison, which will include a greater number of national metrology institutes worldwide under the Mutual Recognition Arrangement of the International Committee of Weights and Measures - (CIPM-MRA) [6].

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9636-29, Session 10

Electron-excited energy dispersive x-ray spectrometry in the variable pressure scanning electron microscope: it's not microanalysis anymore! *(Invited Paper)*

Dale E. Newbury, Nicholas W. M. Ritchie, National Institute of Standards and Technology (United States)

Quantitative elemental microanalysis by scanning electron microscopy/ energy dispersive x-ray spectrometry (SEM/EDS) as performed in a conventional high vacuum (chamber pressure < 10 mPa) instrument is capable of micrometer to sub-micrometer lateral and depth resolution, depending on the beam energy and the composition of the target [1]. The actual analytical footprint is a convolution of the incident beam diameter and electron scattering within the target, which is usually dominant and can be estimated by the Kanaya-Okayama range:

R_(K-O) ("nm")=27.6 (A/[p Z]^0.89)[E_0]^1.67-[E_C]^1.67] (1)

A is the atomic weight (g/mol), Z is the atomic number, ? is the density (g/cm3), E0 is the incident beam energy (keV), and Ec is the shell ionization energy (keV) [2]. The majority of SEMs sold today are capable of variable pressure operation (VPSEM), in which the specimen chamber pressure can be selected in the range 1 to 2500 Pa, providing for discharging of insulating specimens through gas ionization as well as allowing operation with wet specimens or other controlled environmental situations. EDS x-ray spectra can be readily collected during VPSEM operation, suggesting that VPSEM microanalysis is possible, but the spatial resolution that can be achieved is significantly degraded compared to the same beam conditions in a conventional high vacuum SEM. As the beam passes through the elevated pressure region of the specimen chamber, beam electrons are gradually removed from the focused beam through elastic scattering events with gas atoms [2]. Although the majority of these elastic events create small scattering angles, since the length of the gas path is typically 1 – 10 mm, the gas-scattered electrons deviate far out of the focused beam, landing from tens to hundreds of micrometers away. The EDS spectrum measured under VPSEM conditions is always a composite of the x-rays generated by the electrons that remain in the focused beam and strike the selected target along with the x-rays generated where the remotely scattered electrons land. Depending on the pressure and path length, the contribution of these remotely scattered electrons can constitute 90% or more of the measured spectrum. While formulae derived from the Ideal Gas Law and elastic scattering cross sections can estimate the extent the limits of the gas scattering skirt [2], Monte Carlo electron trajectory simulation, such as that embedded in NIST DTSA-II, provides the detailed gas-scattering profile that strikes the specimen and can directly simulate mixed composition targets, e.g., a particle on a substrate [3]. The impact of gas scattering at the major (C >

0.1 mass fraction), minor ($0.01 \leq C \leq 0.1$), and trace ($C < 0.01$) constituent level can be estimated.

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9636-30, Session 10

Development of "water window" laser generated plasma soft x-ray source for contact microscopy experiments

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Visualizing small objects in nanometer scale with high resolution is very important from the point of view of modern science and technology [1-2]. This requires the use of short wavelength radiations, such as soft X-rays and EUV which are capable of resolving nanometer scale features and allowing to capture images with significantly improved resolution [3]. Soft X-ray radiation sources capable of delivering sufficient photon flux in the "water window" (2.3 nm to 4.4 nm) spectral range are also required for imaging of biological specimens within an extremely short exposure time with natural contrast. Soft X-rays can be produced efficiently by large-scale facilities such as synchrotron radiation sources or free electron lasers. These devices are the state of art designed for cutting-edge experiments; however, the limited access to such sources encouraged the development of alternative laboratory soft X-ray sources to open the possibility to perform experiments without the necessity to use the large photon facilities. In this study, a compact table-top laser driven plasma soft X-ray source, developed for contact microscopy experiments, is presented. The source is based on a double-stream gas puff target, irradiated with nanosecond laser pulses from commercial Nd: YAG laser (EKSPLA), which generates laser pulses of 4 ns time duration and energy up to 740 mJ at 10 Hz repetition rate. The source is optimized for maximum emission of soft X-rays in the "water window" (280 – 540 eV) employing pure gases (argon, nitrogen and krypton) and spectral filtering in order to achieve high contrast imaging. Results of the source characterization measurements and dosimetry of the produced soft X-rays are presented and discussed. The potential application of the source for contact microscopy imaging technique is demonstrated by exposing different biological samples and nanoparticles. Results obtained from contact microscopy experiments showed that the high intensity and short pulse soft X-rays produced by laser-plasma allow to capture images of biological specimens and nanoparticles with high resolution, a few tens of nanometers.

References

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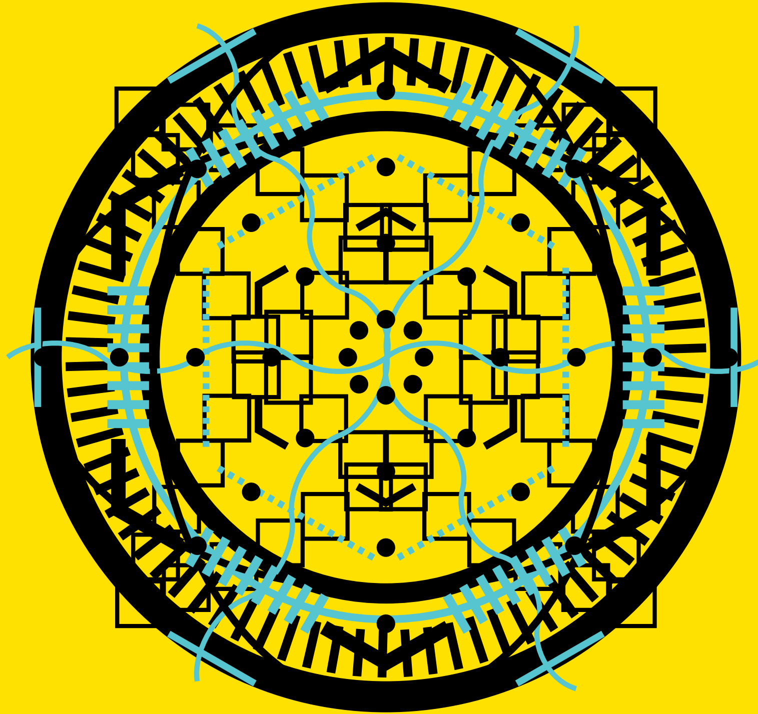

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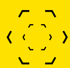
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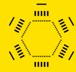
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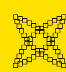
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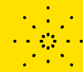
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