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An Overview of Recent Advances in Metrology Development for Nanoelectronics at the National Institute of Standards and Technology (1)

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Nanoelectronics require the introduction of several new uncharacterized material(s) combinations and new processing techniques. The critical metrology and characterization needs of the nanoelectronics industry are being addressed with a broad range of development programs at the National Institute of Standards and Technology (NIST). In this paper, we provide an overview of the recent advances in nanoelectronics enabling metrology developments at NIST.

We will discuss a broad range of new techniques, equipment and infrastructure that are being developed to enable atomic and nano-scale measurements. Examples include, but are not limited to: high resolution, low noise techniques and infrastructure for nanoscale chemical analysis; metrology for characterizing device and material intrinsic reliability; electrical, optical and physical characterization of novel materials; and monitoring of processes.

Introduction

The microelectronics / nanoelectronics industry supplies vital components to the electronics industry and to the global economy, enabling rapid improvements in productivity and the emergence of new high technology growth industries such as electronic commerce and biotechnology. NIST in fulfilling its mission of strengthening the U.S. economy, works with industry to develop and apply technology, measurements and standards, and applies substantial efforts on behalf of the semiconductor industry and its infrastructure. This report describes some of the many projects being conducted at NIST that constitute that effort.

By the late 1980s, the National Bureau of Standards (NBS, now NIST) recognized that the semiconductor industry was applying a much wider range of science and engineering technology than the existing NBS program was designed to cover. The necessary expertise existed at NBS, but spread over many parts of the organization. At the same time, roadmaps developed by the U.S. Semiconductor Industry Association (SIA) independently identified the broad technological coverage and growing industrial needs for semiconductor metrology development. The National Semiconductor Metrology Program (NSMP) was created as a result of US Congressional action. In 1988, the US congress changed the name from NBS to NIST and in 1991, NIST established the Office of Microelectronics Programs (OMP) to coordinate and fund metrological research and development across the agency, and to provide the industry with easy single point access to NIST's widespread projects.

The NSMP has stimulated a greater interest in semiconductor metrology, motivating most of NIST's laboratories to launch additional projects of their own and to cost-share OMP-funded projects. The current NSMP portfolio comprises of a collection of projects under the following broad topics of interest to the microelectronics / nanoelectronic industries:

- Lithography Metrology
- Critical Dimension and Overlay Metrology
- Front End Processing Metrology
- Interconnect and Packaging Metrology
- Process Metrology
- Analysis Tools and Techniques
- Device Design and Characterization
- System Design and Test Metrology
- Manufacturing Support

Most, but not all, of these projects are partially funded by the OMP, which is providing a $12 million budget in fiscal year 2007. Furthermore, in support of the development of nanoelectronics, the President's American Competitiveness Initiative (ACI) enabled by NIST to issue a grant for $2.76M to the Nanotechnology Research Initiative, under the Semiconductor Research Corporation (SRC) for "Beyond CMOS" research, to identify the materials and device architecture to replace CMOS in the 2012 time frame.

Nanoscience involves atomic and molecular level systems and processes that require fundamental understanding of the various physical phenomena associated with very small systems, which behave very differently than systems containing bulk materials with well characterized properties. To complicate matters, nanotechnology entails the integration of nanoscale materials and structures into larger materials components, systems, and architectures. This involves the creation and use of structures, devices, and systems that have novel properties and functions, much different from the bulk properties which depend critically on the size. Thus, control of matter and processes at the atomic and molecular level is essential. For us to have any idea of what is going on in these nanosystems, we need to rely on "measurements" (hence the emphasis on metrology – the science of measurements).

Measurements are an integral part of research, process development, and manufacturing. The microelectronics industry relies very heavily on measurements in process control. Engineers often use the word *metrology* to describe procedures, such as critical dimension measurements, which routinely monitor lithography processes inside the cleanroom. Others generalize it to all in-line measurements. The characterization measurements address quality assurance of incoming materials, wafer screening methods, control and monitoring of equipment and manufacturing processes, diagnostic and failure analysis, and end device production in light of intended design and function. They are best described as a wide range of interdisciplinary activities that determine the structure, composition, properties, and performance of materials and devices, and the relationships among them. The extension of metrology from the microscale to nanoscale science and technology is non trivial because of the differences in responses of the two system scales to external stimuli.

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The role of the NIST in the development of nanosystems has been to provide the basis for understanding differences and harmonizing the experimental results. This is accomplished by assessing the quality of results in the public domain and predicting novel outcomes. For these activities NIST has to:

- Develop new measurements and standards; this entails the developments of methods, techniques, instruments and tools, reference materials and traceability
- Critically evaluate data and understand the foundation for models and simulations to predict properties and function of materials, as well as produce scaleable models and tools for validation

In addition to this paper, the following eight detailed reports on NIST metrology development activities are presented in this volume:

- The Impact of the Dielectric/Semiconductor Interface on Microstructure and Charge Carrier Transport in High-Performance Polythiophene Transistors **Y. Jung, R. Kline, E. Lin, D. Fischer et al.**
- The Challenge of Measuring Defects in Nanoscale Dielectrics **K. P. Cheung and J. Suehle**
- Superconformal Film Growth: Mechanism and Quantification **T. Moffat, D. Wheeler and D. Josell**
- In Situ Gas Phase Diagnostics for Hafnium Oxide Atomic Layer Deposition **J. E. Maslar, W. Hurst, D. Burgess, W. Kimes, N. V. Nguyen and E. Moore**
- Combinatorial Methodology for the Exploration of Metal Gate Electrodes on HfO_2 for the Advanced Gate Stack **K. Chang, M. Green, J. Suehle, J. Hattrick-Simpers et al.**
- Interface Barrier Determination by Internal Photoemission: Applications to Metal/Oxide/Semiconductor Structure **N. V. Nguyen, O. Kirillov, W. Jiang, J. Maslar, W. Kimes and J. Suehle**
- Scanning Probe Microscopy for Dielectric and Metal Characterization **J. J. Kopanski**

In this paper we present high level reviews of a few of the over 150 nanosystems-related metrology activities (other than those presented in this volume) currently underway at NIST. The reviews are presented in the form of case studies.

Case Studies

Case Study #1: Metrology for Deep Ultraviolet (UV) Lithography

John H. Burnett, Simon G. Kaplan, Eric L. Shirley, Deane Horowitz, and Eric Benck at NIST have developed the most accurate optical properties measurement capability in the world that resulted in the "rediscovery" of an intrinsic birefringence (IBR) at short wavelengths in crystalline materials which were presumed to be isotropic and non-birefringent (2).

Lithographic systems for nanosystems will require lenses of high-index materials, with indices greater than 1.8 at the illumination wavelength. The materials will also have to have a low value of IBR, less than about 30 nm/cm. However, as shown by NIST

measurements indicated in the figure below, most high-index UV materials have IBR values too high.

Based on NIST's measurements (see Figure 1), the only candidates that can meet the specifications are ceramic spinel and the garnets: pyrope and lutetium aluminum garnet (LuAG). Consequently, lithographic materials manufacturers have explored the possibility of developing these materials. It has been found that only LuAG shows reasonable prospect for manufacturability to lithographic material standards, and industry efforts have now been focused on developing LuAG as a lens material for advanced lithography tools.

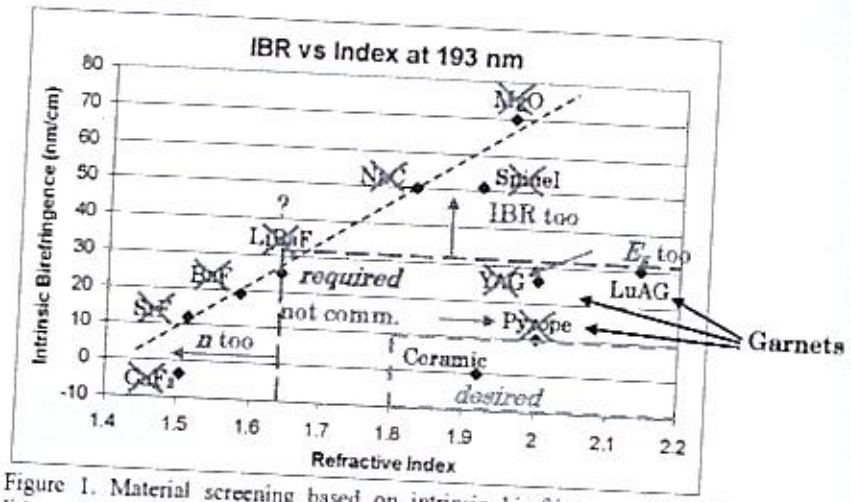

Figure 1. Material screening based on intrinsic birefringence (IBR) for high-index lithographic stepper lens materials

Case Study #2: Critical Dimension and Overlay Metrology

New methods for critical dimension (CD) measurements are needed to enable the detailed characterization of nanoscale structures produced in the semiconductor industry and for nanotechnology applications. Small angle x-ray scattering (SAXS) measurements with synchrotron sources have shown promise in meeting several grand challenges for CD metrology. However, it is not practical to depend upon x-ray synchrotron sources, which are large national facilities with limitations in the number of available instruments. To address this problem, a laboratory scale SAXS instrument for critical dimension measurements on periodic nanoscale patterns has been developed, designed, installed, and tested by Wen-Li Wu et al.(3). The system possesses two configurations, SAXS and ultra-small-angle x-ray scattering (USAXS), with a radiation target of either copper or molybdenum. With these configurations, the instrument is capable of accessing scattering angles that probe length scales ranging from ca. 0.5 nm to 0.2 μm. This technique leverages the fact that silicon is transparent to x-rays for E > 13 keV. Figure 2 below shows the CD-SAXS instrument at NIST in the SAXS-Mo configuration with monochromator.

The NIST CD-SAXS measurements have been benchmarked successfully against a synchrotron-based SAXS at the Advanced Photon Source of the Argonne National Laboratory. The results from standard line/space gratings possessing periodic line-space patterns with CDs of tens to hundreds of nanometers show that the laboratory-scale system can quantitatively measure parameters, such as the pitch, linewidth, height, linewidth roughness, and sidewall angle. These results show that laboratory-scale measurements are feasible and can be used for research and development purposes or to assist calibration of optical scatterometry and CD-scanning electron microscopy instruments.

The advantages of the CD-SAXS technique include the facts that it is a non-destructive, requires no sample preparation and is capable of measuring pattern cross section as shown schematically in Figure 3. This technique uses optical scatterometry targets with a typical beam spot size 100um x 100um and is ideal for sub-45 nm structures at sub-nm precision in pitch and linewidth. As a matter of fact, this X-ray measurement becomes easier with smaller structures. The technique can measure *"2-D" and buried patterns e.g.*, via posts, pads, etc made of all materials including metals, polymers and dielectrics. The primary limitation of the measurement is that the data collection rate is too slow for production metrology because of the significantly low x-ray beam fluxes currently available from today's non-synchrotron x-ray source.

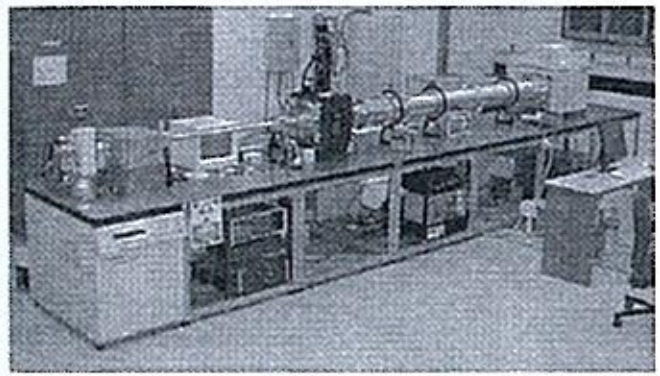

Figure 2. Photograph of the CD-SAXS instrument at NIST in the SAXS-Mo configuration with monochromator.

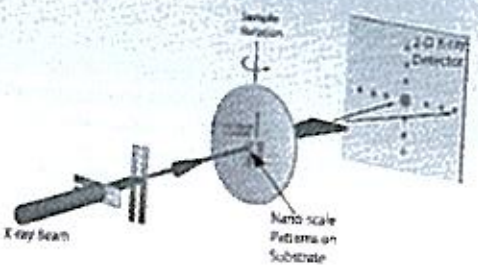

Figure 3. An illustration sample-beam arrangement of the SAXS in transmission configuration

Case Study #3: High Resolution Microcalorimeter X-Ray Spectrometer for Chemical Analysis

Microcalorimeters are a novel alternative technology for energy-dispersive X-ray spectroscopy on scanning electron microscopes (SEMs). The energy resolution of microcalorimeters is 10 to 50 times better than existing Si-based X-ray sensors. Consequently, microcalorimeters can easily resolve closely spaced X-ray lines that cannot presently be distinguished. The resolution of microcalorimeters is even good enough to distinguish chemical shifts, thus providing chemical as well as elemental information.

The improved resolution of microcalorimeters enables the analysis of nanometer scale films and particles. Nanoscale structures are best probed by weakly penetrating, low energy electron beams that produce X-rays in the ~1 keV range where there are numerous line overlaps. These overlaps are easily resolved by the microcalorimeter. X-ray spectra from a microcalorimeter and a conventional X-ray sensor are compared in Figure 4.

The exquisite sensitivity of microcalorimeters is derived from their typical operating temperature of 0.1 K. NIST has devoted considerable effort to make this ultralow temperature accessible in a straightforward and cost-effective manner. Cooling from 3 to 4 K to 0.1 K is performed using a NIST-designed two-stage adiabatic demagnetization refrigerator. Cooling from 300 K to 3 to 4 K was originally performed using liquid nitrogen and helium. Recently, however, NIST has designed a refrigerator system where liquid cryogens are replaced by a closed-cycle cryocooler. The only consumable for this refrigerator is electricity.

It is critical that the refrigerator used to cool the microcalorimeter leave SEM performance unchanged. In particular, vibrations from the cryocooler must not degrade image quality. NIST has successfully worked to minimize the vibration signature of the cryocooler. SEM images taken with and without the cryocooler in operation show that the cryocooler mounted on the SEM has almost no effect on image quality.

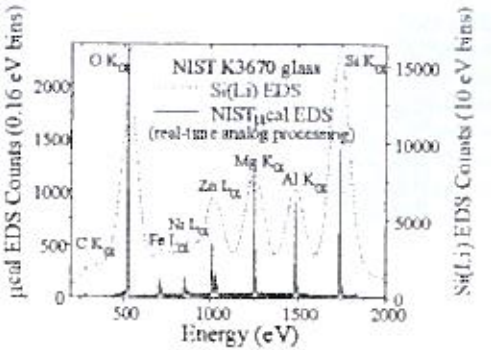

Figure 4. Comparison of X-ray spectra from a microcalorimeter and a conventional X-ray sensor of NIST K3570 reference glass.

To move microcalorimeter technology from the laboratory to commercial availability, NIST is assisting STAR Cryoelectronics, a Santa Fe New Mexico based company, develop a commercial microcalorimeter spectrometer. The STAR prototype unit is shown in Figure 5. STAR has successfully fabricated high resolution X-ray microcalorimeters and is preparing for a full system demonstration.

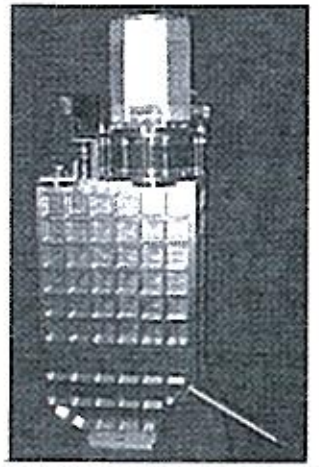

Figure 5. Commercial Microcalorimeter X-ray spectrometer under development by STAR Cryoelectronics for use on scanning electron microscopes. The microcalorimeter sensors are located at the end of the cold finger at bottom right.

Case Study #4: Spectroscopic characterization of buried metal/dielectric interfaces through IR-transparent substrates and thin films.

One of the biggest impediments in the study of novel materials is investigating the chemical reactions that are occurring at buried interfaces. Often with the addition of a top metal electrode, annealing, or other processing steps, interfacial chemical reactions

give rise to new and unique electrical properties, such as the creation of defects, shorts, or other electrically active material. The challenge then becomes correlating this electrical change with physical and chemical changes to ultimately obtain the desired properties. Christina Hacker, Lee Richter, and Curt Richter have developed a vibrational spectroscopy technique that takes advantage of the transparent nature of the silicon substrate in the infrared to examine the interfacial chemical reactions that occur in molecular electronic and high-k dielectric samples. This technique, unlike conventional optical and electronic spectroscopies, does not require a thin metal top electrode, enabling one to study the full range of processing conditions and use the same samples for both electronic and spectroscopic measurements with less sample contamination.

Within molecular electronics, the self-assembled monolayer and underlying substrate are very well known because they have been studied using a wealth of surface science techniques. However, the structure of the organic monolayer is unknown after deposition of the top metal electrode because most surface science probes lose sensitivity when the metal layer approaches 10nm or less. Instead, researchers often assume that the monolayers before and after metallization are identical. This naive assumption is fundamentally flawed because mush chemistry occurs during the deposition of metal, that often changes the structure of the molecules, creates new chemically reactive species, and even results in simple mechanical shorting of the electronic device. Obviously, there is a need to characterize the materials within the molecular electronic device in order to adequately correlate the electronic transport properties of molecular structures.

The vibrational spectroscopy technique developed by researchers in the Semiconductor Electronics Division at NIST is known as P-polarized backside reflection absorption infrared spectroscopy (pb-RAIRS or backside FTIR, See Figure 6 below). This nondestructive technique takes advantage of an IR transparent substrate and IR mirror to obtain chemical (composition) and conformational information (atomic arrangements) at the buried interface and has been used at NIST to study bonding of metal with molecular monolayers and high-K dielectrics(4). This work has allowed us to characterize dielectrics under full metallization and to use identical samples for FTIR and electrical characterization.

Two examples of the type of information that has been received from pb-RAIRS investigation of molecular monolayers and high-k materials on silicon are shown below in Figures 7 and 8. First, aliphatic chains tethered to silicon consisting of 18 CH_2 subunits were assembled on both silicon and silicon oxide substrates. However, the electrical measurements of monolayers on silicon continued to show no molecular characteristics. Using pb-RAIRS and investigating the monolayers under 200 nm of metal, we observed that the monolayers directly attached to silicon were displaced with metallization under Al, Au, and Ti(5) (Figure 7). Further exploration determined the cause for the molecular displacement was reaction of the metal with the substrate to form a silicide and remove the monolayers(6). Vibrational spectra observed monolayers under Ag, a metal which does not form a silicide, as shown in Figure 7. In addition, a soft methylene mode near 2800 cm^{-1} was observed indicative of some metal "fingering" through the organic monolayer, in agreement with the electrical results on identical samples.

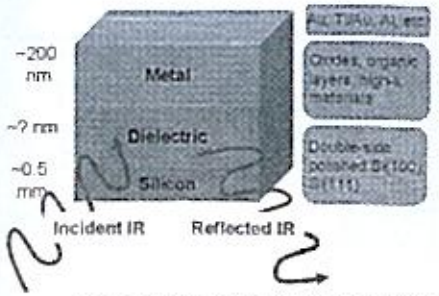

Figure 6. Schematic of a pb-RAIRS sample

Second, introducing the novel materials systems of high-k dielectrics and metal gates necessitated optimizing a lot of parameters to achieve the desired electrical properties. Often electrical properties, such as work function, would change with processing with little to no understanding of the origin of these phenomena. NIST and SEMATECH researchers worked closely to correlate the changes in chemical and conformational structure with the corresponding changes in the electrical response of these materials.

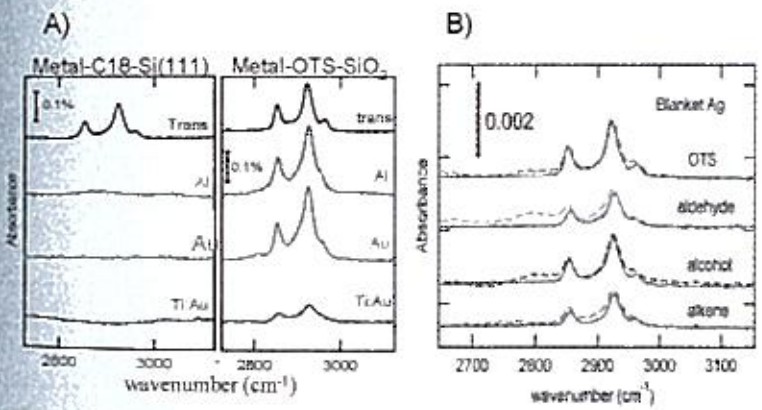

Figure 7. A) pb-RAIRS spectra of aliphatic monolayers on silicon (left) and silicon oxide (right) for Al, Au, and Ti top metal electrodes. B) pb-RAIRS spectra of aliphatic monolayers on silicon (aldehyde, alcohol, alkene) and silicon oxide (OTS) as a function of covalent bonding atom. OTS, aldehyde, and alcohol species bond via a Si-O-C bond and the alkene functional group attaches through a Si-C covalent bond.

Figure 8 below shows the pb-RAIRS observation of a chemical reaction at the HfN-HfSiOx interface as a function of annealing conditions (7).

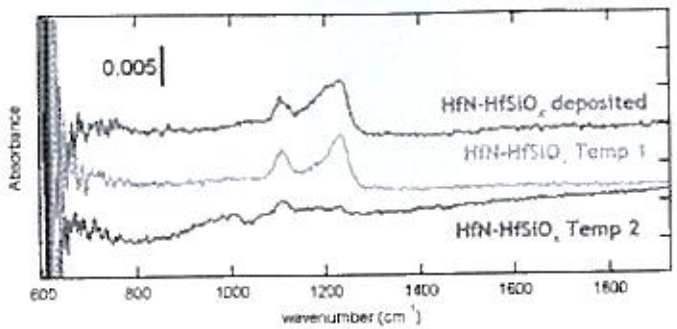

Figure 8. pb-RAIRS spectra of silicon-HfSiO$_x$-HfN samples (prepared by Sematech) as a function of annealing temperature. The data show removal of silicon oxide with annealing in agreement with electrical data.

Case Study #5: Spin-Transfer Microwave Nano-Oscillators and Memory Elements

Bill Rippard, Matt Pufall, Shehzaad Kaka, Stephen Russek, and Tom Silva at NIST-Boulder have developed metrology to measure and understand spin-based device behaviors at microwave frequencies and nanometer length scales (8). The devices are based on the concepts of spin-momentum transfer, where the coherent transfer of angular momentum between spin polarized electrons and a ferromagnetic metal has led to an entirely new way to control and manipulate magnetism, namely one which does not involve a magnetic field. The spin transfer effect has been used to switch the states of nanoscale storage elements, such as those used for magnetic random access memory (MRAM) applications, as well as to induce coherent magnetization precession (having a quality factors, Q values, greater than 10,000) in these devices. The potential future applications of this technique include reference oscillators, directional transmitters and receivers in cell phones and radar systems, nano-wireless communications within or between chips, high-frequency signal processors, on-chip microwave spectroscopy, switching of MRAM structures, and new spin-based logic concepts.

In terms of switching in magnetic storage devices, the spin transfer effect is very promising in future applications since the strength of the interaction becomes stronger as device dimensions are decreased. The magnitude of a magnetic field generally scales as the inverse of the device dimension. However, the magnitude of the spin transfer effect scales as the inverse of the square of the device dimension, since it is proportional to the current density. Because of this scaling, it is expected that spin transfer switching of MRAM devices will be more efficient than conventional (field based) switching below the 75 nm node. The metrologies developed at NIST are focused on comparing the theoretical models for switching in these devices to the experimentally determined switching behaviors. As a metrology institution, particular attention is being paid to devising new methods to measure the parameters that are theoretically expected to determine switching thresholds and then to compare the measured thresholds to those predicted. In this regard they have explicitly confirmed aspects of the methods used to compare theoretical work considered only at zero temperature to measurements performed at room temperature as well as pointed out the shortcomings in doing so.

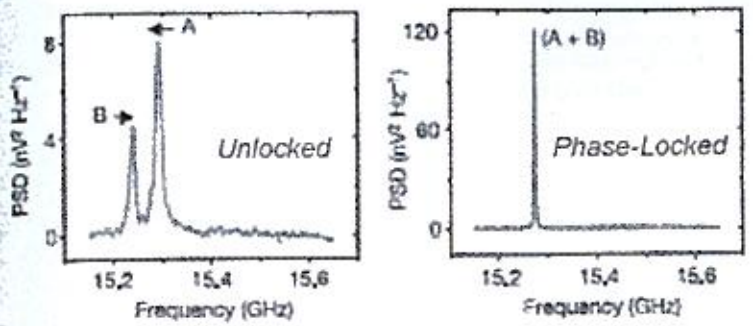

Figure 9. Phase-locking of the dynamic *magnetic properties of two spin-transfer oscillators located 500nM apart.*

The researchers have also developed methods to directly probe the microwave dynamics that are induced by the spin transfer interaction. This effect has led to the realization of a new spectrometer that is tunable from a few hundred megahertz to over 40 gigahertz, depending on the applied dc current and magnetic field, and is only 50nM in diameter. The developed metrologies have allowed them to probe the details of the magnetic excitations and design devices that have the highest Q-values reported. Building on these techniques, they have shown that groups of magnetic oscillators synchronize their individual 10nW signals to achieve signal strengths equal to the square of the number of devices involved. This represents a direct avenue towards increasing the power output of the devices to commercially viable levels and additionally improve upon the spectral purity of the output, as shown in Figure 9.

Concluding Remarks

Looking forward, significant metrology development will be required to further enable nanosystems. Specifically, the following metrologies are needed (9):

- Nondestructive 3D imaging of embedded interface, nanostructure and atomic scale matrix properties
- Optical properties of isolated and integrated low dimensional materials
- Integrated metrology and modeling tools that deconvolve probe-sample interactions
- Nanoparticle monitors for ES&H including size, dose and composition.

Also, the following generic metrology-related issues must be addressed in order to take full advantage of the available measurement technologies:

- Improved signal to noise ratio
- Reduced or no measurement induced sample damage
- Elimination of surface contamination or denaturing from sample handling, ambient contamination, etc.
- Eliminate or minimize sample preparation.

The goals of many of the metrology programs at NIST are to provide these capabilities. The objective of this collection of presentation is to inform the nanoelectronincs industry about these programs. We would also welcome feedback, where appropriate, from the industry. Submit your feedback on NIST's semiconductor nanoelectronics projects at http://www.eeel.nist.gov/omp/survey.pdf.

Acknowledgements

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