

Metrological SPMs for quantitative traced measurements in the nanoscale

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Outline:

- 1. Need for quantitative dimensional determination at the nanoscale
- 2. Measurements at the nanoscale
- 3. How to get quantitative measurements
- 4. Traceability matters
- 5. SPMs
- 6. Calibration of SPMs and transfer standards
- 7. Metrological SPM at CEM



Metrology topics at the ITRS 2009 Metrology roadmap

microscopy - critical dimension (CD) and overlay - film thickness and profile - materials and contamination analysis - dopant profile - in situ sensors and cluster stations for process control - reference materials - correlation of physical and electrical measurements - packaging.

According to ITRS 2009 Report

- Critical dimension measurement with nm level precision is difficult to achieve.
- A variation in features size of 1/10 of the nominal dimension often results in significant changes in device properties.
- The fundamental challenge for factory metrology will be the measurement and control of atomic dimensions while maintaining profitable high volume manufacturing.
- Long-term research into nano-devices may provide both new measurement methods and potential test vehicles for metrology.



		2010	2012	2014	2016	2018
	Flash 1/2 pitch (nm)	32	25	20	16	13
	DRAM 1/2 Pitch (nm)		36	28	23	18.0
	MPU Printed Gate Length (nm)		31	25	20.0	16.0
	MPU Physical Gate Length (nm)	27	22	18.0	15.0	13.0
	Wafer Overlay Control (nm) - 20% DRAM	9.0	7.1	5.7	4.5	3.6
	Wafer Overlay Control Double Patterning (nm)	6	5	4	3	3
	Lithography Metrology					
Gate	Physical CD Control (nm) Allowed Litho Variance = 3/4 Total Variance	2.8	2.3	1.9	1.6	1.3
	Wafer CD metrology tool uncertainty (3 ¹ , nm) at P/T = 0.2	0.55	0.46	0.37	0.31	0.26
	Etched Gate Line Width Roughness (nm) <8% of CD	2.1	1.8	1.4	1.2	1.0
Dense Lines	Printed CD Control (nm) Allowed Litho Variance = 3/4 Total Variance	3.3	2.6	2.1	1.7	1.3
	Wafer CD metrology tool uncertainty (3s, nm) at P/T = 0.2	0.7	0.6	0.5	0.4	0.3
	Double Patterning Overlay Metrology					
	Double Exposure and Etch - Process Range (nm)	6.4	5.1	4.0	3.2	2.5
	Double Exposure and Etch - Uncertainty (nm)	1.3	1.0	0.8	0.6	0.5
	Spacer PEE process					
	First pass CD control (after etch) - Process Variation (nm)	3.0	2.4	1.9	1.6	1.3
	First pass CD control (after etch) - Uncertainty (nm)	0.6	0.5	0.4	0.3	0.3
	Front End Processes Metrology					
	High Performance Logic EOT equivalent oxide thickness (EOT), nm	0.65	0.5	0.5	0.5	0.5
	Logic Dielectric EOT Precision 3/ mm	0.0026	0.002	0.002	0.002	0.002
	Interconnect Metrology					
	Barrier layer thick (nm)	3.3	2.4	1.7	1.3	1.1
	Void Size for 1% Voiding in Cu Lines	4.5	3.6	2.8	2.3	
	Detection of Killer Pores at (nm) size	4.5	3.6	2.8	2.3	

Manufacturable solutions exist, and are being optimized

Manufacturable solutions are known

Interim solutions are known

Manufacturable solutions are NOT known

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Example: ITRS 2009 Metrology Roadmap

Replacing the

silicon dioxide with

a low-к dielectric of

the same thickness

reduces parasitic

switching speeds and lower heat

capacitance, enabling faster

dissipation

Existing Challenges

- Measurement Gap Sidewall barrier thickness and sidewall damage (compositional changes in low k)
- New Porous low k is projected for 22 nm ¹/₂ Pitch
- Detection of Voids after electroplating
- Monolayer interface for new barrier-low k

Air Gap sacrificial layer does not require unique metrology

- Metrology is needed for 3D Integration
 - TSV Depth and Profile through multiple layers
 - Alignment of chips for stacking wafer level integration
 - Bond strength
 - Defects in bonding
 - Damage to metal layers
 - Defects in vias between wafers
 - Through Si via is high aspect ratio CD issue
 - Wafer thickness and TTV after thinning
 - Defects after thinning including wafer edge
- Emerging / Gated Interconnects
 - Native Deice Interconnects



1. Need for quantitative dimensional measurements



Interconnection Metrology



Example: ITRS 2009 Metrology Roadmap

Front End Process (FEP) Metrology

3D Metrology – Complex structure measurement and inspection are required

e.g. high A/R holes, film thickness & properties on sidewall



Pipe-shaped BiCS Flash Memory (R. Katsumata, Toshiba)



TCAT (Terabit Cell Array Transistor) (J. Jang, Samsung)



Nanomedicine

- a) Direct measurement of different elements in the nanoscale: cells, receptors, membranes, organelles, proteins, biomolecules
- b) Topographical study of surfaces
- c) New developments in this discipline (ref. K.K. Jain, Med Princ Pract 2008; 17:89-101)
- **Nanodiagnostics:** Molecular (and Nanomolecular) diagnostics as the nanothechnology-on-a-chip for total chemical analysis systems, Imaging with nanoparticle contrast materials, Nanobiosensors
- Nanopharmaceuticals: Nanotechnology-based drugs, Targeted drug delivery to site of action, Drug delivery from implanted nanopumps and nanocoated stents
- Reconstructive surgery: Tissue engineering with nanobiotechnology scaffolds, Implantation of rejection-resistant artificial tissues and organs
- Nanorobotics: Vascular surgery by nanorobots introduced into the vascular system, Remote controlled nanorobots for detection and destruction of cancer
- Nanosurgery: Tools for nanosurgery, Nanosensors implanted in catheters to provide real-time data during surgery, Nanolaser surgery



Chemical Nanometrology

Three priority areas to meet industrial needs:

- measurement of the chemical composition of thin films and concentration of species.
- characterisation of structural properties.
- granulometry of nanoparticles in different media (liquids, gases).

Challenging areas for surface chemical and structural analysis: (identified by the Surface Analysis Working Group at the CCQM/BIPM)

- microelectronics (key driver of metrology and standardisation developments in surface analysis for the last forty years)
 - life sciences, bio-nano-objects;
 - manufactured nano-objects.

New certified reference materials useful for chemical analysis are needed.

Source: Co-Nanomet



Thin Films

Large application and economic impact over a wide range of industry sectors:

- As buried layers, core to the performance of state-of-art microelectronics and magnetic data storage devices.

- As surface functional layers, employed for wear-, impact- and scratch-resistance, friction-control, lubrication, anti-reflection coatings, bio-activity or passivation, wettability and easy-to-clean or self-cleaning surfaces.

Generic measurement tasks: Thickness, chemical composition, structure, conformity, uniformity and integrity, surfaces and interfaces (chemistry, structure, roughness, interdiffusion, etc.), impurities and dopants, mechanical properties, ... Techniques available in the sub-100 nm range:

- length-based methods, potentially traceable to the SI unit of length, and
- areal-mass based methods, potentially traceable to mass or mole (per unit area)

Source: Co-Nanomet



Nanomechanics

Key technology for the support of important industrial sectors as thin films and coatings in industrial applications. Also important for the development of micro and nanotechnology devices and components.

Most advanced areas: nanoindentation and AFM indentation measurements



Source: Co-Nanomet

1. Need for quantitative dimensional measurements

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Tendencies and Challenges in nanometrology



Co-Nanomet, A. J. Pidduck, L. Koenders, T. Dziomba, L. E. Depero and M. Gee









Estimation (1st) and **Reduction** (2nd) of *U* should be applied to all measurement and characterization techniques used in nanometrology

- laser interferometry (visible and X-ray),
- laser Doppler,
- inductive and capacitive sensors, piezoelectrics (PZTs),
- ellipsometry, variable-angle spectroscopic ellipsometry (VASE),
- optical spectroscopy
- optical microscopy: Nomarski, total internal reflection microscopy (TIRM), confocal, ...
- contact profilometry,
- total integrated and angle-resolved scatterometry (TIS, ARS),
- STM, AFM, SNOM, ...
- X-ray diffraction,
- electronic and diffraction microscopy (XTEM, STEM, SEM, HRSEM),
- Raman spectroscopy and Fourier transform infrared absorption,
- Auger (AES), X-ray photoelectron (SPS) & backscattering (RBS) spectroscopies,
- X-ray fluorescence
- .../...







Dimensional Nanometrology Needs

Increasing the lateral scanning range of AFMs to several tens of millimetres to allow the measurement of large area structured surfaces, wafers and optics.

(higher scanning speed, intelligent probing and control systems, sampling strategies including combining SPMs with other instrumentation, intelligent multi-sensor concepts, improved data fusion algorithms, etc.)

Improved SPM resolution (<u>repeatability</u> & <u>reproducibility</u>) to allow the measurement of smaller structures with higher accuracy. Better theoretical understanding of surfaceprobe interactions and refined probe characterization for a true quantification of dimensional measurements.

Source: Co-Nanomet



Dimensional Nanometrology (cont.)

Increasing true 3D-measurements: Semiconductor industry needs to measure high aspect ratio (HAR) structures and critical dimensions (CDs). Development of MEMS and NEMS.

Development/Improvement of new 3D nano machines (nano CMMs)

Appropriate traceability infrastructure (development of metrological AFMs, transfer artefacts, optical interferometers, ...).

Fast and accurate areal measurement and characterisation techniques (technical specifications and standards coming soon).

Source: Co-Nanomet





If Traceability Chain too long, Uncertainty too high Need of direct link to SI unit (1 to 2 steps)





Some applications of SPMs

- precision engineering (e. g. form/waviness/roughness, surface finish, film/layer thickness; products & tools)
- micro-fabrication (e. g. sensors, optical gratings, micro-lenses, micro-sieves, MEMS; master & product)
- **semiconductor fabrication** (e. g. critical dimension CD, pitch, line edge roughness LER, overlay; masks & wafers)
- **material science** (e. g. grain size distribution, roughness, hardness/nano-indentation, force spectroscopy)
- crystallography, solid state physics (arrangement of atoms, molecules, clusters)
- chemistry (e.g. polymer research: elasticity, local chemical sensitivity and dispersion)
- life sciences (features of tissue and local chemical activity, often studied in liquids)
- nano-science/nano-engineering (nanostructured or nanometrically functionalized surfaces, nanoparticles, nanorods, carbon nanotubes -CNT, etc.)
- Calibration of reference structures (standards) for other measurement techniques

	GOBIERNO DE INDUSTRIA, TURISMO Y COMERCIO
	SPM Categories, attending to its Traceability
A	Reference SPMs with integrated laser interferometers. Often referred to as "metrological SPMs" (no Abbe error). Direct Traceability, via the wavelength of the laser used, to the SI unit of length.
В	 SPMs with integrated sensors for positioning monitoring, e.g. capacitive and inductive sensors, strain gauges, encoders. <u>Calibrated</u> either by temporarily attaching laser interferometers to the scan or by using high-quality physical transfer standards. Two subcategories are distinguished: B1. Active position, tracking to the scheduled position by means of a closed- loop, usually called "closed –loop" configuration. B2. Includes position measurements but without closed loop.
С	SPMs in which the position is determined from the electrical voltage applied to the adjusting elements. <u>Calibration</u> usually with physical transfer standards.
5. SPM	E. Prieto - CEM ImagineNano 2011 - Industrial Forum



SPM Main Elements					
casing / mounting	mechanical, acoustic, electromagnetic, thermal characteristics				
sample with sample holder	object under study				
x-y coarse positioning	rotational deviations				
x-y scanner	to displace the probe or alternatively the sample	C, B, A			
z coarse approach	to decrease distance between the probe and the sample up to a value where the z actuator can operate	С, В, А			
z actuator	element to keep the probe under constant conditions while scanning the sample				
probe	tip to investigate sample properties	C, B, A			
detector loop	element to detect small changes in the property under study (as a photodiode)	С, В, А			
x-y and/or z displacement transducer	encoders, capacitive or inductive displacement transducers, strain gauges				
closed loop	active position control	B1			
laser interferometers	position measurement and control (closed loop)				
angular sensors	tilt measurement and control (closed loop)	A			



VDI/VDE 2656-Part1: Determination of geometrical quantities by using of Scanning Probe Microscopes Calibration of measurement systems

Objectives of the guideline:

Comparability of measurements of geometrical quantities by traceability to the unit of length

Definition of minimum requirements for the calibration process and the conditions of acceptance

 Ascertainment of the calibratability (assignment to calibratability categories)

 Fixing of the scope of a calibration (conditions of measurement and environments, ranges of measurement, temporal stability, transferability)

 Provision of a model according to GUM to calculate the uncertainty for simple geometrical quantities in measurements using a scanning probe microscope

Definition of the requirements for a result report

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PRELIMINARY CHARACTERIZATION OF THE MEASURING SYSTEM

(prior to the calibration process)

Study of the waiting time before putting into operation (warm-up, initial drift)

Waiting time after change of sample / probe or other interventions to reach stable conditions

Noise of the system is one of the limiting factors of the metrological capabilities of an SPM. •Investigate z-noise by using two measuring modes: standstill (scan off) and record z signal, and dynamic mode (with scanning motion) on a flat surface, scan the same line twice and take the difference. Scan speed, scan range and measurement rate should be set to typical values.

• x-y noise is usually of lesser importance than z noise. It can be investigated using a sample with straight edge or a line with small step height, aligning the edge of the sample to one axis (for example y axis) and doing the scan in the x axis and checking by means of successively recorded profiles the variation of the location of the edge.

Guidance deviations can be identified by measurement at standards or with additional equipment (as autocollimators). For example out of z plane can be investigated by using a flat standard with a well defined reference area calibrated by interference microscope. Subtraction of the reference data from the actual SPM data yields that information. By repeating this process it is possible to distinguish between temporary effects or permanent cross-talk.

Evaluation of the effect of ambient conditions (changes in temperature, humidity, air flow, mechanical and acoustic vibrations, electromagnetic interferences, etc,)

Long time stability (reproducibility)



Verification and calibration of SPM with samples and standards





Aim of the SPM calibration: determination of the calibration factors C_x , C_y , C_z However...a total of 21 deviations or degrees of freedom can be identified (as in any 3D coordinate measuring machine)

3	positional deviations (non linearities)	xtx, yty, ztz
6	straightness deviations (underlined cross-talk of lateral scan movemements to vertical z axis)	xty, <u>xtz</u> , ytx, <u>ytz</u> , xtx, zty
9	rotational deviations	xrx, xry, xrz yrx, yry, yrz zrx, zry, zrz
3	rectangularity deviations	ywx, zwx, zwy



	To determine	Type of Standard	Explanation	Section
C _z	Calibration of the z axis	Step height standard	Step height measurement,	6.5, 6.6
ztz	Non-linearity z axis	Set of step height standards	linearity	
C _x C _y xtx yty	Calibration of the x axis Calibration of the y axis Non-linearity x axis Non-linearity y axis	1D or 2D lateral standard	Pitch measurement, rotation, linearity	6.4, 6.6
xty ytx	Cross talk of the lateral axes	2D lateral standard	Pitch measurement, rotation, linearity	6.4, 6.6
xtz ytz	Cross talk of the lateral movements to the z axis	Flatness standard	Guidance error (out of plane: temporary drift or scanner bow)	6.3
ywx	Rectangularity deviation	2D standard	Angle formed by the two axes on orthogonal structures	6.4, 6.6
zwx zwy	Rectangularity deviation	3D standard	Angle formed by the two axes on orthogonal structures	6.4, 6.6



LATERAL CALIBRATION

★By interferometers

★By Physical Standards

The **periods** of the standards to be used should be selected according to the measurement purpose and to the evaluation method available. It is important not only the selection of the scan range but also the scan speed or the measurement rate as the calibration factors are strongly influenced by dynamic non linearities and image distortions.

With 1D standards the calibration factors can be determined only succesively in **two measurements** with the standards being rotated by 90°.





For 2D structures it is possible determine in to one measurement the calibration factors and the nonorthogonality of the grating γ_{xy} (angle formed by the px period in the x direction of the grating, and p_v vectors differing from 90°)





LATERAL CALIBRATION

Evaluation methods

Centre of gravity (GC)

In the local space

To determine for example p_{xmeas} : fit two paralell regression lines g_0 (close to the left of the image) and g_n (close to the right) through columns of grating structures. Rise a vertical line s_x to g_0 and g_n lines. Determine p_{xmeas} by dividing the distance of the points of intersection of s_x with g_0 and g_n by the number of enclosed grating periods

Fast Fourier Transform (FFT)

In the Fourier space

Determination of the measurand mean grating periods by measurement of the position of the appurtenant peak





- > Lateral standards allow to determine also **distorsions** in the measured image
- > Depending on their origin these errors are **temporary**, **permanent** or **dynamic**



6. Calibration of SPMs and standards



VERTICAL CALIBRATION

The elongation of the z piezo can in some systems be directly measured **by laser interferometry**. The calibration is done by applying voltage externally to the z-actuator and recording simultaneosuly the signal from the interferometer. **Non linearity** of the z piezo scan can be studied by increasing the voltage in equal steps. The **dynamics of the z scan** can be investigated by changing the duration of the voltage steps.

Physical standards, step height standards, are easier to handle than optical interferometers but the traceability chain is longer and the uncertainty of the transfer standard must be added to the uncertainty of the SPM calibration. In order to account for the **nonlinearities** of the z scan it is necessary to use a **set of standards** of different height.





Vertical movements must follow the surface topography and are usually much faster than lateral scan movement, so **z-axis dynamic properties play an important role**.



VERTICAL CALIBRATION

Methods of data analysis

Hystogram method

The sample tilt has to be previously substracted. In the histogram plot of the height value distribution it must be identified the two peaks corresponding to the two height levels. The center of gravity in each of these two peaks is determined and their difference is calculated.



ISO 5436

Application is done by fitting a line through the central part (Wm) of a positive or negative bar. As reference two equally sized sections (Ws) on both sides of the bar are selected with equal distance to the edges of the bar (We), and a line is also fitted through these values. In this way the edges are not taken into consideration. The height of the bar is defined as the normal distance of both lines.



6. Calibration of SPMs and standards



Main calibration standards





Different kinds of standards

(<u>www.nanoscale.de</u>)



6. Calibration of SPMs and standards





Metrological large range SPM at CEM

7. Metrological SPM at CEM

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MAIN FEATURES

✤ Three dimensional positioning and measurement of samples in the range 25 mm x 25 mm x 5 mm

Solution Sol

Three dimensional Abbe offset free design, intersection of measuring beams at contact point of the probe sensor (offset less than 0.1 mm)

♥ Resolution below 0.1 nm



INTEGRATION OF TWO SYSTEMS

- Nanopositioning and nanomeasuring machine
 NMM-1 from SIOS
- SPM Probe *Dualscope DS-95-50-E from DME*

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NMM SET UP

- 1) Metrology frame: Zerodur frame mounted on a granite foundation
- 2) Interferometry and traceability: three miniature interferometers (one for each axis) with HeNe stabilized lasers. The measuring mirrors are put together forming a corner mirror structure with the outer reflecting surfaces. The sample object is placed on the base plate of the corner mirror. Calibration of the z scanner against the z interferometer is done previous to measuring.
- **3)** Actuation and guidance: 3D stacked arrangement (x, y and z on top) for the guide driving system. The position of the corner mirror is measured by the interferometers and the signals are used as control variables to avoid translatory errors. Two angular sensors and four z drivers keep the angles of the z stage constant.
- **4) Electronics control:** NMM stage control coarse surface slopes and holds SPM iin the middle of its measuring range, SPM probe system act as zero point indicator.



Basic set-up of the NMM machine: 1 x-interferometer, 2 y-interferometer, 3 z-interferometer, 4 Zerodur® frame, 5 Roll and yaw angle sensor, 6 Pitch and yaw sensor, 7 probe system, 8 Measuring object, 9 Corner mirror, 10 Foundation, guide-drive systems of the 11 x-axis, 12 y-axis 13 z-axis

NMM-1, SIOS



SPM

- **1) Scan range** 50 μm x 50 μm x 5 μm
- When coupled to NMM, the movement is restricted to 2.7 µm in z direction
- 3) Coarse approach to the sample is done with piezo motors to use the z range of the scanner optimally.
- 4) Different **operation modes**: DC (contact and non contact) AC, STM
- 5) Scanner with a **built-in linearization sensor** calibrated against the z axis interferometer



DS-95-50-E, DME



NMIs with metrological SPMs

		NMI	Country	Range	Status		
		Europe – S	SFM in service				
		DFM	Denmark	70 x 70 x 6 µm ³	In service		
		METAS	Switzerland	400 x 70 x 5 μm ³	In service		
		NPL	United Kingdom	100 x 100 x 5 μm ³	In service		
		PTB	Germany	25 x 25 x 5 mm ³	In service		
		Europe – S	SFM under construct				
	\rightarrow	CEM	Spain	25 x 25 x 5 mm ³			
		CMI	Czech Republic	200 x 200 x 10 µm ³	1 st comparison (height, pitch)		
		INRIM	Italy	100 x100 x 15 μm³	2 nd comparison (height, pitch)		
arge		MIKES	Finland	100 x 100 x 16 μm ³	1 st comparison (height, pitch)		
kange		LNE	France	300 x 300 mm ² x 50 μm			
		VSL	Netherlands	100 x 100 x 20 μm ³			
		Non European region					
		NIST	United States	50 x 50 x 5 µm ³	In service		
		NMIJ	Japan	100 x 100 x 12 µm ³	In service		
		NMC/A*S TAR	Singapore	25 x 25 x 5 mm ³	1 st comparison (height, pitch)		
		<u>.</u>	:		· · ·		

7. Metrological SPM at CEM



Conclusions:

- 1. There is a need for quantitative measurements at the nanoscale
- 2. Many properties depend on Critical Dimensions
- 3. Traceability is a crucial matter. The shorter the chain to SI unit, the lower the uncertainty
- 4. Calibration standards needed for maintaining the performance of SPMs and other instrumentation
- 5. CEM and other NMIs developing metrological instruments and offering low-*U* calibration services to R&D and industry
- 6. No Metrology (infrastructure), no Development
- 7. Researchers should become Metrologists too



Thank you for your attention

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