Shallow Probe metrology

Addressing the challenges in elemental composition, thickness determination, and dopant dosimetry from FE to BE

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ABSTRACT

New materials and processes are steadily introduced in semiconductor technology to solve some new challenges of device performance. In the future, some choices will follow the ITRS nodes directives while others will be manufacturer- and/or product-dependent. These different varied orientations make the situation more and more complex for metrology tools. They need to be versatile enough to adapt relatively quickly to new varied material selections and be able to efficiently support the process engineers in the tough ramping-up phases as well as to ensure continuous yield improvement. In the metrology domains of monitoring elemental composition and thickness in layers/multilayers and dopant dosimetry, CAMECA developed an original solution based on the LEXES technique.

Introduction

In CMOS fabrication, scaling down is accompanied by the introduction of new materials to achieve better performance [1]. The important changes are not limited to gate stacks and the development of high-k materials and electrodes, and include new capacitor dielectrics, interconnect, metal contacts, plugs, barriers, and dopant techniques. For gate oxide metrology, even though the thickness range tends to sub-nanometer starting from the 45nm node, there is still a variety of thickness ranges to be investigated. The substrates can also be varied and may encompass Si-based, SiGe-based, SOI and strained-Si substrates. These different environments require metrology evolution. This evolution needs to combine an increasing complexity in metrology of composition, thickness and profilometry with the constraints of the fab production environment such as repeatability, reproducibility, stability, pattern size, Through-put (T-put), particle contamination and reliability. Additionally, the ideal metrology tool is non-contact and non-destructive and would allow the injection of the wafer back to the process. The challenge is getting more complex with the measurement of these combined new layers of different thicknesses. These stacks can vary from a few tens of Å of different layers for flash applications, a real buried layer of thin high-k oxide under the metal gate to control directly the mobility effect, or SiON under a poly-Si layer to be used at gate electrode.

X-ray-based techniques

Traditionally, optical techniques such as ellipsometry have addressed several thickness issues. But the technique is becoming restricted, as a large number of parameters impacting the refractive indices are coming along with the integration of new materials: crystalline state, strain, process techniques, and in some cases composition. In addition, metal layers are challenging for these traditional techniques. Therefore, even though optical techniques allow reaching high analysis T-put, other tools based on X-ray techniques are now present in semiconductor metrology [2]. There are three well-known X-ray based techniques in the metrology of composition and thickness: XRR (X-ray reflectivity), XRF (X-ray fluorescence) and XPS (X-ray photoelectron spectroscopy) [2,3]. XRR is a strong technique for thickness measurement, but despite the fact that no references are needed, accuracy strongly depends on the model to adapt to graded layer, interface and roughness (in a way similar to ellipsometry). Additionally, similar densities of buried materials cannot be distinguished [2]. XRF requires high-quality standards, and long integration times might also be needed depending on the yield and consequently low T-put. XPS is a reference analytical technique but is limited to ultra-thin layers (max. 7 to 10nm) and requires complex algorithms to be able to process the data. Integrating such powerful analytical lab techniques into

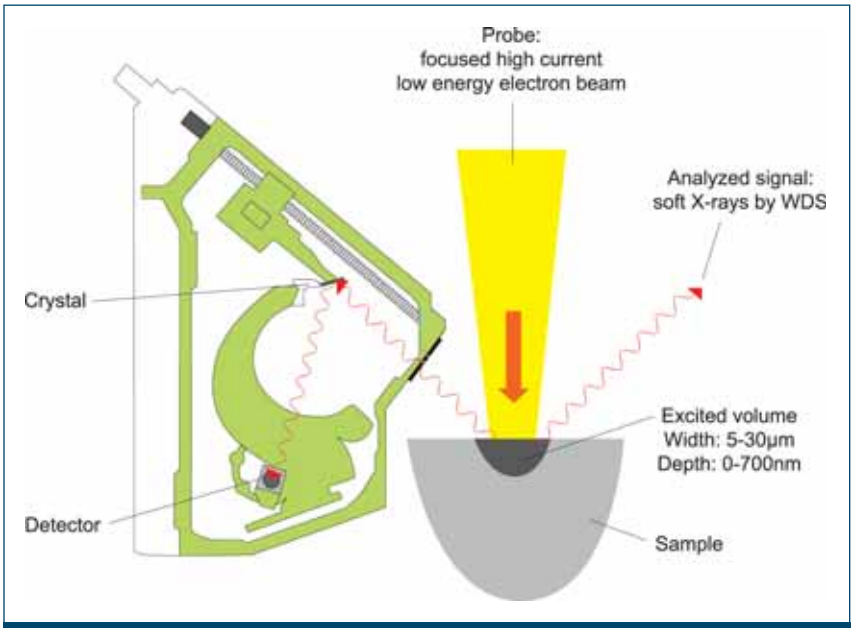

Figure 1. Schematic of Low energy Electron induced X-ray Emission Spectroscopy (LEXES) with a WDS detection system.

TABLE 1: SOME PERFORMANCES ON TYPICAL APPLICATIONS

Applications	Silicon oxynitride	SiGe	High-k	Metal Nitride	Implants in Si
	SiON	SiGe	$HfO_2/Al_2O_3/HfO_2$	TiN, TaN, WN	E.g. As
Detection Limit (at/cm^2)	For N and O 2×10^{14}at/cm^2	Ge: 5×10^{13}	Hf: 5×10^{13} Al: 5×10^{13}	5×10^{13}	5×10^{13}
Dose repeatability For 1σ %	<1 % for N & O dose >5×10^{15} at/cm^2	<1 % for Ge dose >5×10^{16}	<1% for Hf >1.5×10^{16}at/cm^2 and Al > 2.2×10^{15}at/cm^2	<1% for doses > 5×10^{16}	<0.5% for dose >1×10^{15}at/cm^2
Throughput wph (for 5pts)	>4 for N >2×10^{15}at/cm^2	>5 for film > 100 nm	<7 for Hf >1.5×10^{16}at/cm^2 and Al > 2.2×10^{15}at/cm^2	>5 for film > 100 nm	>6 for dose > 1×10^{15} at/cm^2

metrology is a challenge in itself: it is not guaranteed that the metrology tool will provide as much information as the analytical tools even though they are based on the same technique.

In order to take up the challenge of monitoring composition, thickness and dopant levels in this materials-issue-rich metrology environment, CAMECA has developed a new tool family, the Shallow Probe, based on a less known X-ray based technique called LEXES (Low energy Electron induced X-ray Emission Spectroscopy). Shallow Probe systems are now in operation in numerous fabs around the world addressing routinely varied issues to follow process development. After a brief introduction of the LEXES technique, several application examples will be presented on different materials characterized with the CAMECA Shallow Probe metrology tool.

LEXES: a unique surface probing technique

The Shallow Probe tool is a simple, original and unique tool based on the LEXES technique, also sometimes called EXES [4,5]. The material is probed by a low energy electron beam and the soft X-ray emission is collected in WDS (Wavelength Dispersive Spectroscopy) spectrometers (Figure 1). The technology is similar to CAMECA's EPMA (Electron Probe Microanalysis) instrument, but a low-energy, high-current electron column has been specifically designed by CAMECA to optimize surface analysis instead of bulk analysis. The probing energy can vary from a few hundred eV to 10keV and the electron beam current can be adjusted from 0.1 to 100µA. The resulting probing volume depth, as shown in Figure 1, is typically down to 700nm. The beam size can be adjusted from 5 to 60µm according to the application (blanket or patterned wafer).

Three WDS spectrometers are available. Each spectrometer is composed of a moveable tuning crystal selecting X-ray photons of a given wavelength and counted by means of a gas flow detector. Different types of crystals can be mounted in one spectrometer. The configuration is chosen depending on the elements to be analyzed. Simultaneous measurements can be performed for different elements in the same layer for higher T-put. The system allows accurate, repeatable and stable control of all elements from Be to U and is an ideal versatile tool to monitor different multilayer processes from Front-end (FE) to Back-end (BE). The development in conjunction with world-class fabs and its large installed base makes the Shallow Probe a fully fab-compatible tool with two 300mm load-ports, patterned wafers analysis capabilities, and easy-to-use interface.

The elemental analysis is direct and consists of measuring the Peak (P) minus Background (B) intensity for a specific wavelength characteristic of a given species. This P-B intensity is proportional to the dose of this species. The sensitivity factor used to convert the P-B intensity into a dose is determined from a reference sample. The composition of the layers is directly measured element per element. The performance will depend on the crystal configuration chosen. Typical performances are listed in Table 1. The dose values (at/cm^2) only *casual* in ion implantation can be converted into concentration (at%) and thickness (nm) using either the material density or the multi-energy model. Multi-incident energy measurements provide information about the in-depth structure of the specimen. Data thus collected is treated via a model developed at CAMECA and allows the refinement of elemental analysis, or gathering of specific depth information. One typical example is the simultaneous monitoring of the composition (Ge at%) and the SiGe thickness in SiGe layers. The tool is extensively used to monitor N dose and thickness in SiON layers, implant dosimetry in Si for Ultra-Shallow Junction or in SiGe, and composition/thickness in varied layers such as oxides or metals.

Silicon oxynitride materials

Oxynitride layers have been used since the late 1990s and are becoming highly engineered. The Shallow Probe has been used for several years to control N doses in typically ~20Å-thick layers [6]. The technique allows both O dose (signal is strong, no limitation) and N dose control down to the mid e14at/cm^2 range (mapping shown in Figure 2). The repeatability, measured as nine points in a line, is typically lower than 1% for doses as low as 5e15 at/cm^2 dose. If lower doses are monitored, the repeatability becomes higher.

A new challenge in SiON metrology is that process engineers now require the dose monitoring to be performed on the SiON capped with a poly-Si gate. With the Shallow Probe, it is possible to monitor O, N and thickness with similar performances as for non-capped up to ~40nm thickness poly on top of an SiON layer. In Table 2, the repeatability performances are shown for SiON with a 10nm capping. The non-uniformity of N dose across the 300mm wafers in that case is much higher at around 4 to 6%. Typically, the measurement stability for the N dose (range 2e15at/cm^2) and the thickness is 1.3 to 1.5% over seven days for these capped layers. SiON under higher poly gate capping (<100nm) can be analyzed, but the N sensitivity will be degraded because of X-ray emission absorption by the poly-Si. Therefore, CAMECA is working on the integration of new crystals to improve N detection and obtain better performances for capped and non-capped SiON layers. For an N-

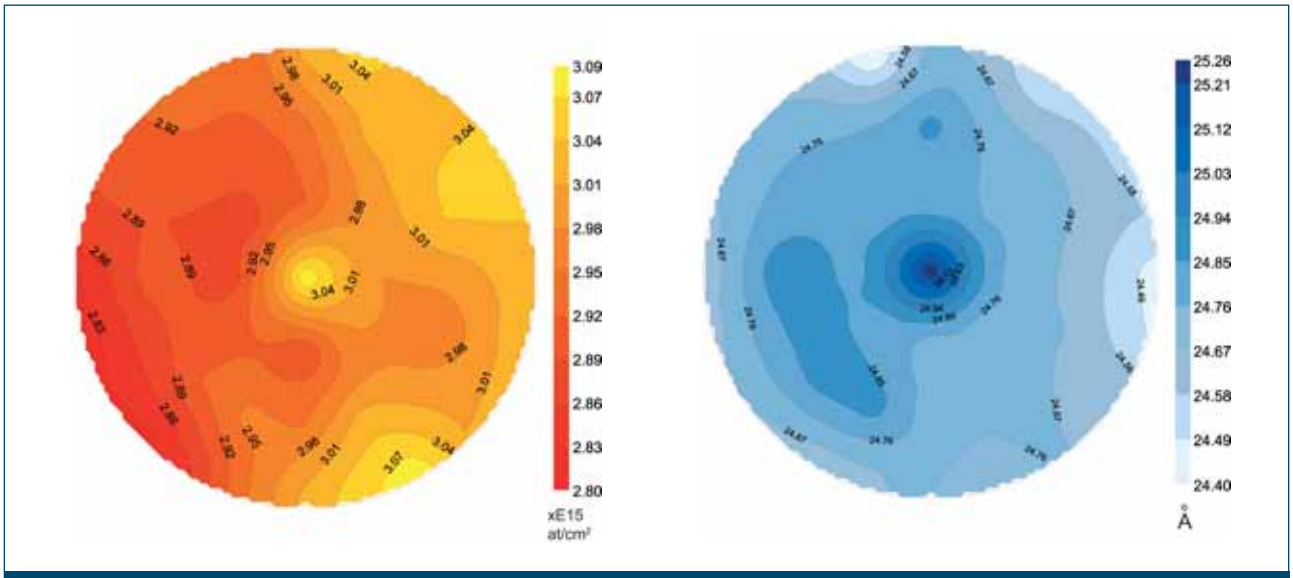

Figure 2. Typical mapping of (a) N dose and (b) thickness monitoring in SiON layers with the Shallow Probe.

dose of 7e14at/cm² (un-capped layers), a repeatability of 1.1% (1σ) is obtained with new crystals instead of 2.2% with our current crystals.

Oxides

Oxides are used in gate stacks, flash and DRAM materials. For these different processes, the metrology challenge is the same for the Shallow Probe in terms of elemental and thickness determination in varying multilayer sequences of different materials. Several measurements on oxides are presented below.

Thin layers

Thin layers of Atomic Layer Deposition (ALD) HfAlO have been investigated on two different substrates (Si/SiN and Si/TiN) and three growth processes (A to C) are implemented. The three elements Hf, Al, and O have been monitored to determine composition and thickness (Table 3). The trend of the nominal value (increasing Hf content from A to C) between the three processes is well measured on both types of substrates. The total thickness of the HfAlO films on SiN substrates is 5Å whereas it is slightly thicker on TiN (~6.75Å). The repeatability of the measurements is around 0.5% for Hf and Al and between 1 and 2% for O.

TABLE 2: REPEATABILITY ON NINE POINTS FOR FIVE SION WAFERS CAPPED WITH 10NM POLY-Si GATE. A COMPOSITION OF 17 AT% CORRESPONDS IN THIS CASE TO A DOSE OF 2E15AT/CM²

Wafer	N Conc. (at%)	RSD on at%	Thickness (A)	RSD thickness
1	16.75	0.75	20.43	0.44
2	16.9	0.56	20.43	0.64
3	16.16	0.67	20.63	0.48
4	17.11	0.59	20.69	0.36
5	17.46	0.67	19.27	0.52

Thick layers

La_2O_3 oxide is an interesting high-k material and has been investigated in $Hf_xLa_yO_z$ layers deposited by ALD. Both Hf and La have been analyzed in a series of five wafers, which are made by ALD cycles: H1L1 (one Hf cycle for one La cycle). The composition has been independently measured by RBS. LEXES shows an excellent correlation with RBS as indicated in Figure 3. A positive offset of the LEXES data relative to RBS data is observed; this offset increases for low La content. It is in agreement with the fact that in RBS both Hf and La peaks are partially overlapping due to poor energy resolution. Therefore, with a strong Hf signal and going towards a low La content and large Hf content like in H10L1, the La peak is barely detected, resulting in a growing error in composition estimation in RBS. The advantage of the LEXES technique is that when certain transitions overlap (Mα for Hf and La in this specific case), a different transition can be chosen (Mξ in this case).

TABLE 3: COMPOSITION AND THICKNESS DETERMINATION OF THIN HfAlO LAYERS (REP = REPEATABILITY)

Wafer	Hf at%	Hf Rep RSD%	Al at%	Al Rep RSD%	O at%	O Rep RSD%	Hf/Al ratio	Thickness (A) Al_2O_3	Thickness (A) HfO_2
1 – Process A on SiN	13.61	0.5	24.1	0.47	61.63	1.52	0.55	2.59	2.41
2 – Process B on SiN	20.5	0.38	15.6	0.33	63.76	1.66	1.3	1.56	3.42
3 – Process C on SiN	24.84	0.29	10.16	0.42	65.01	2.17	2.45	0.96	3.98
4 – Process A on TiN	14.41	0.27	23.45	1.07	62.13	0.39	0.61	3.31	3.43
5 – Process B on TiN	22.45	0.41	14.26	0.52	63.29	1.19	1.57	1.86	4.99
6 – Process C on TiN	26.17	0.56	9.32	0.48	64.51	1.12	2.81	1.17	5.58

Composition and thickness determination

ALD ZrO_2 layers have been investigated in a series of 3 wafers. One part of the first wafer was coated with a thin ~20nm ALD TaN layer. The composition determination was performed and the estimated thickness compared to SIMS data. Results presented in Table 4 show that there is a slight increase in the Zr/O ratio as a function of thickness, and also an excellent agreement on the thickness estimation (less than 5%) between SIMS and LEXES. The measurement of such layers is relatively challenging in SIMS because it requires suitable reference samples for the thickness and concentration calibration and the analysis T-put is far lower.

In-depth information and energy spectral resolution

On the same HfLaO layers as shown in Figure 3, the multi-energy signal from LEXES allows the extraction of in-depth information from samples. Application examples for SiO_2/HfO_2 stacks have already been researched and published [4]. This article presents results for HfLaO. Figure 4(a) shows the intensity of La Mξ line as a function of the energy, where the intensity profile as a function of the energy indicates the depth of the layer. The first qualitative analysis of the energy curves shows that the La is further buried for films with higher Hf ALD cycles. For the same nominal Hf burst, the depth of the La-rich layer is more important for smaller La nominal conditions, which would be consistent with an assumption of the La dissolution into the Hf layer.

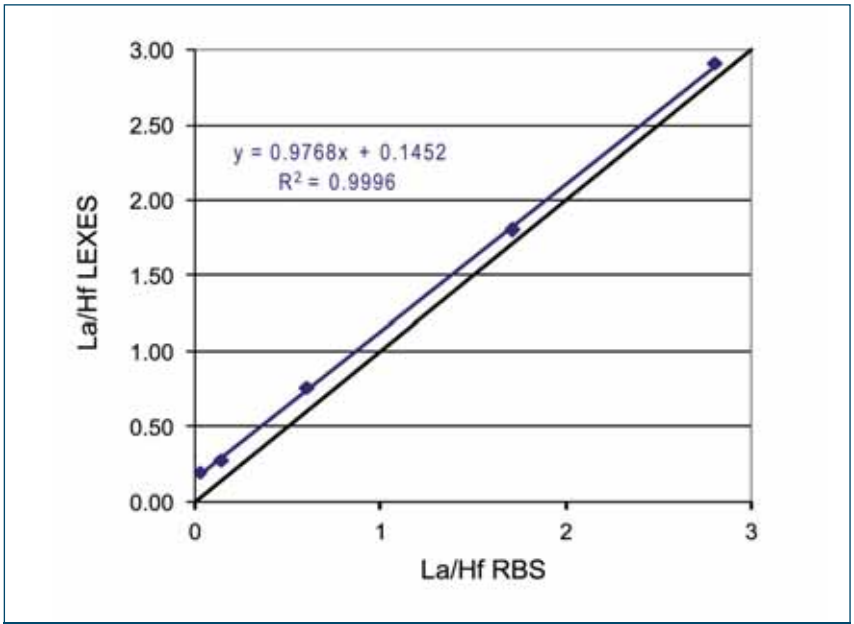

Figure 3. Correlation of LEXES and RBS measurements on composition measurements on $Hf_xLa_yO_z$ layers.

Analyzing the wavelength spectra, two components in the La Mξ peak can be distinguished (Figure 4(b)): one at 19.39Å for surface La and another at 19.34Å for the deepest layers. No large database is available on chemical peak shift for this rare earth element. However, glass containing 12% La_2O_3 were measured in the same conditions and the peak was observed at 19.41Å. These first results seem to indicate that the surface La layer is in an oxidation state close to La_2O_3 and the buried one is close to $Hf_xLa_yO_z$. Further investigations are required to complete this specific study but the LEXES metrology tool has shown capabilities to detect energy shift as small as 1.7 eV and to provide in-depth information.

Precursor contamination measurements

The tool's flexibility allows tracking of contamination in thin films, either by scanning around one specific peak or by collecting of a larger wavelength spectrum to see all the emitting species in that energy range. The spectrum is then similar to EDS (Energy Dispersive Spectroscopy). The identification of unexpected contamination is made possible by the combined full spectrum and spectral resolution capabilities.

Zr contamination in $Hf_xLa_yO_z$ layers from the Hf precursor

Four wavelength dispersive spectra around Zr Lα peak recorded on the HfLaO wafers are overlaid in Figure 5. There is a clear increase in Zr content

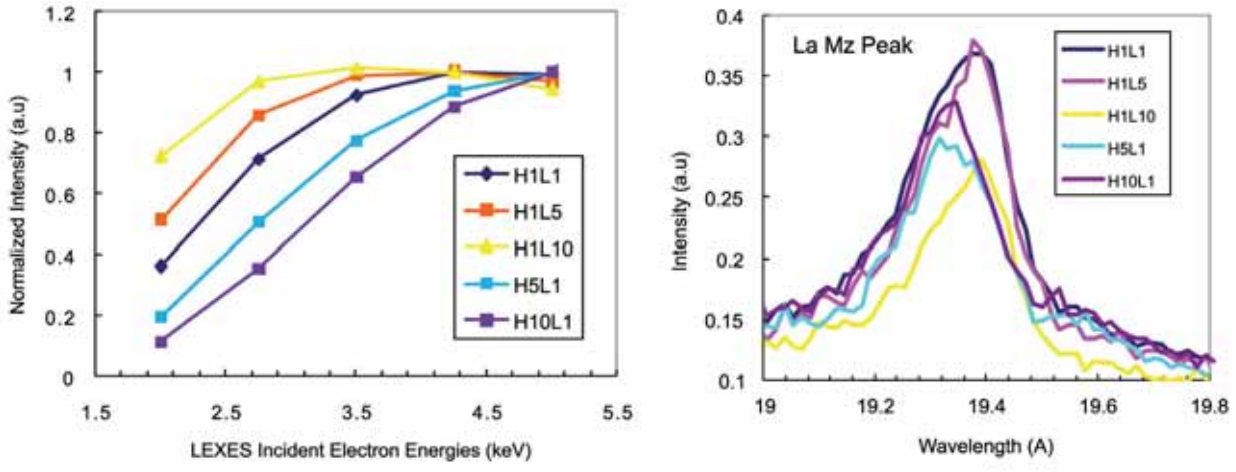

Figure 4. HfLaO layers – La Mξ peak investigation by LEXES: a) Intensity profiles as a function of incident energy; b) Peak position.

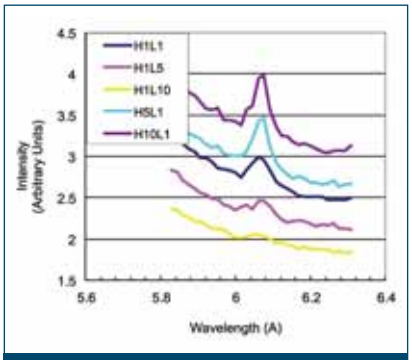

Figure 5. LEXES measurements: Zr peak evolution as a function of Hf cycles. The wafers are the same as those featured in Figure 3.

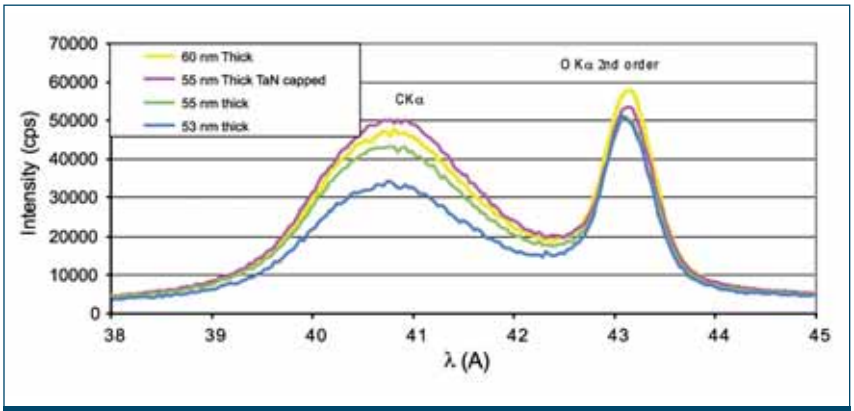

Figure 6. C contamination steadily increasing in ZrO_2 wafers as a function of layer thickness GST layers.

in the HfxLayOz as a function of Hf ALD cycles. This is in agreement with expectations, as it is well known that Hf precursors typically have several hundred ppms of Zr unless specialized purification methods are used. Air Liquide, who kindly provides the HfLaO films, had measured the Zr level in the Hf precursor as 2000 ppm. The Shallow Probe's sensitivity to the detection of impurities of this magnitude is easily demonstrated.

C contamination in ZrO_2 layers

The C contamination of the ZrO_2 wafer series (see Table 4) was measured and is shown in Figure 6. The C dose increases from 10 to 19% (quite non–uniformly across the wafer) for a thickness increase of 10%, while the TaN layer contains a very high additional percentage of C (20 to 30%). Note the wavelength spectral resolution of the Shallow Probe tool allows easy discrimination of Cκα lines from the Okα 2nd order line.

TABLE 4: ZrO_2 WAFER COMPOSITION DETERMINED BY LEXES AND THICKNESS BY LEXES AND SIMS

Wafer	Zr at% LEXES	O at% LEXES	Thickness (nm) LEXES	Thickness (nm) SIMS
1	31.19	68.81	55.40	52.00
2	32.38	67.63	53.00	53.00
3	33.80	66.20	60.40	62.00

TABLE 5: COMPOSITION AND THICKNESS DETERMINATION IN GST LAYERS

Wafer	Ge at%	Sb at%	Te at%	N at%	Thickness (Å)	RSD thickness
1	19.94	19.82	44.92	15.32	736.80	0.88
2	27.05	24.86	48.06	N/A	686.76	0.32
3	25.93	24.08	49.95	N/A	649.10	0.32

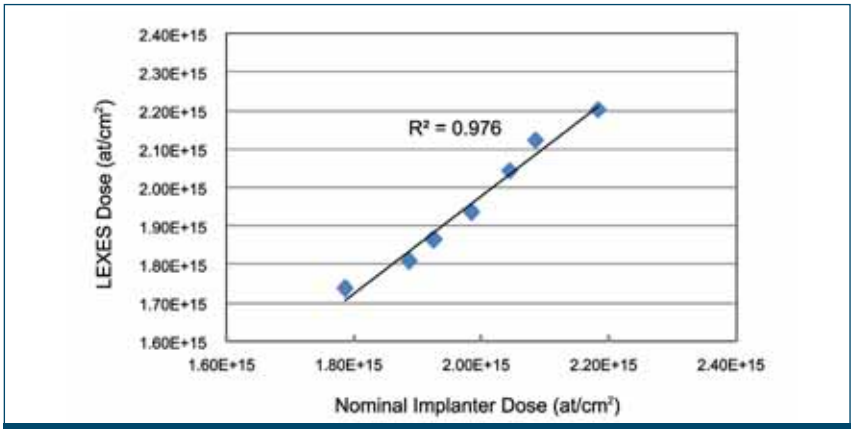

Figure 7. 500eV B implant correlation between nominal implanted dose and LEXES measurements.

GST layers

For PCRAM (Phase-Change RAM) based on the amorphous/crystalline change, GST ($Ge_2Sb_2Te_5$) composition is commonly monitored in fab metrology. To lower the crystallization temperature during growth, N is also added into the layer, resulting in four elements that need to be monitored. An example of composition and thickness determination by the Shallow Probe is shown in Table 5.

Shallow implants

Dose implant metrology by non-electrical techniques can be divided in two main categories: deep implants monitored mainly by SIMS, and shallow implants monitored by surface techniques. With the Shallow Probe, all shallow implants can be monitored over a large range of doses (5e13 to 5e16at/cm^2) used to form Ultra Shallow Junction. The Shallow Probe routinely addresses BPLAD or PPLAD, BDP, PDP, As, B (Figure 7), and P in Si. The measurement is not sensitive to the activation and can be done before or after annealing. The implant energy range that can be monitored is large for As and P (3keV to 65keV) and restricted from 100eV to 5keV for B. The Shallow Probe easily discriminates dose gradations of 5%. Linearity is maintained for both very shallow and deeper implants as shown for As implants in Figure 8(a) shallow implant energy at 4keV and (b) medium implant energy at 60keV.

Conclusions

The various illustrated examples have shown that the Shallow Probe is a versatile tool that can cover a large number of metrology aspects. Issues such as elemental composition,

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• Layer Thickness
• Dopant Dosimetry
• Elemental Composition

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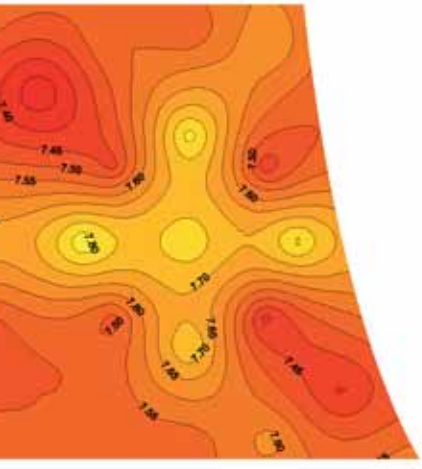

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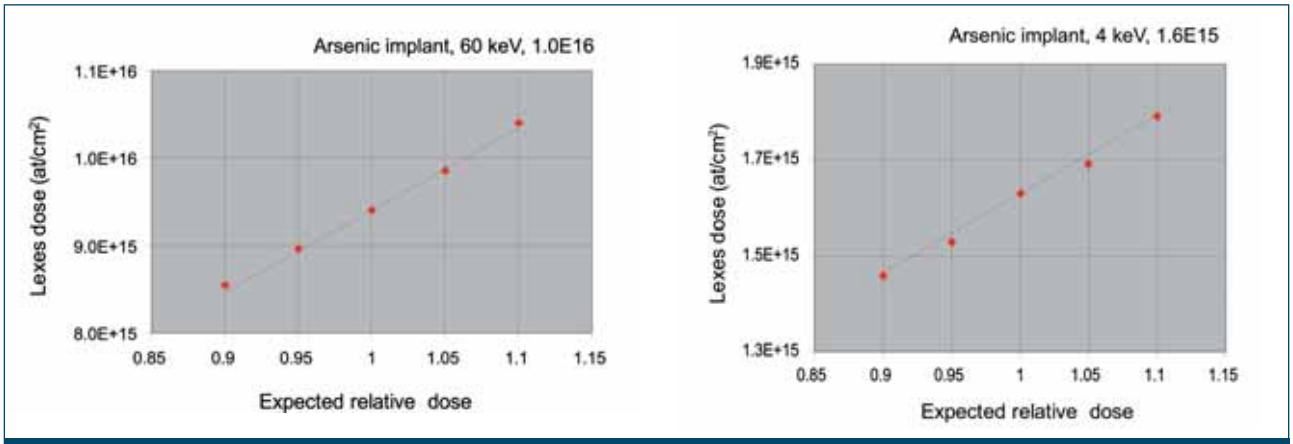

Figure 8. As Shallow Implants: (a) shallow implant energy 4keV for average As dose of 1.6e15at/cm²; (b) medium implant energy 60keV for average dose of 1.06e16at/cm².

thickness, dopant dosimetry, and in-depth information can be addressed for a variety of materials in different environments (substrate, thin or thick multilayers):

- N and O doses in various thermally- and plasma-nitrided silicon oxynitride layers, as processed or capped with a poly layer up to 100nm thick
- oxides layers containing mainly Hf, Zr, Al, La, of a few tens of Å or up to 100nm, uncapped or capped by metal layers
- implant dosimetry on B, As, and P with maintained performance over a large implantation energy.

Measurements on metal layers, thin barrier layers, SiGe, and SiC are also routinely performed in fabs.

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