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The Low Energy X-ray Spectrometry Technique as Applied to Semiconductors

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Abstract: We describe the recent introduction of low energy X-ray emission spectrometry as a metrology technique to control the fabrication process in the integrated circuit industry. The benefits of this particular analytical method and the wide field of potential applications are addressed.

Key words: LEXES, metrology, process control, shallow implants, thin films

Introduction

Low energy X-ray emission spectrometry (LEXES) is based on the same physical phenomenon as electron probe microanalysis (EPMA), that is, the electron-induced core ionization of atoms and the subsequent X-ray emission that is used as an analytical signal. But unlike EPMA, the aim of LEXES is not the analysis of bulk micron-size volumes, but the optimization of near-surface information at the nanometer scale. This involves a quite distinct instrumental design: A LEXES tool requires electron optics capable of transporting 100 times more electron current than EPMA at impact energies as low as only a few hundred electron volts, under very good vacuum, and also requires a specific model to process the low energy data. This is mainly to obtain the sensitivity required for useful metrology on modern semiconductor materials (a good introduction is offered by the International Technology Roadmap for Semiconductors, 2005 edition, available at http://www.itrs.net/Common/2005ITRS/Home2005.htm), including trace (less than 0.1 at.%) elements in ultrathin materials. Figure 1 illustrates the high nitrogen X-ray signals obtained with a 30-μm electron beam spot size from a series of oxy-nitride samples consisting of only 1–2-nm-thick layers on top of silicon, used as a gate dielectric in metal oxide semiconductor (MOS) transistors.

Materials and Methods

Originally developed on a laboratory apparatus (Bonnelle et al., 1994), the LEXES technique was recently implemented on a metrology tool, designed by Cameca and called the Shallow Probe (Staub et al., 2002). This tool consists of an electron column delivering an electron beam with an energy varying between 0.2 and 10 keV, which impinges over the sample surface under a vacuum 1E-8 Torr. X rays emitted by the sample are analyzed thanks to up to three wavelength dispersive spectrometers. According to the tool model, the instrument can accommodate either sample pieces only or a full wafer up to 300 mm in diameter. The instrument is fully automated and controlled through a dedicated graphical user interface, allowing the user either to acquire X-ray spectra by wavelength scanning or to run quantitative analysis on predefined spectral positions corresponding to some elemental emissions. This article is founded on the experience recently acquired with this tool in the semiconductor industry.

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Some Key Metrology Needs in the Modern Semiconductor Industry

Chips Fabrication Lines and Process Control Requirements

Process Tolerance and Precision

Each process step has its own degree of variability within which the performance of the chips will remain under control. This acceptable range of variability is called the process tolerance (T). To efficiently monitor the stability of the process, any metrology tool needs to demonstrate a precision (P) significantly better than T. For most of the process today, this leads to precision requirements *better than 1%* relative to the process target value, implying repeatability and reproducibility values better than 0.2%.

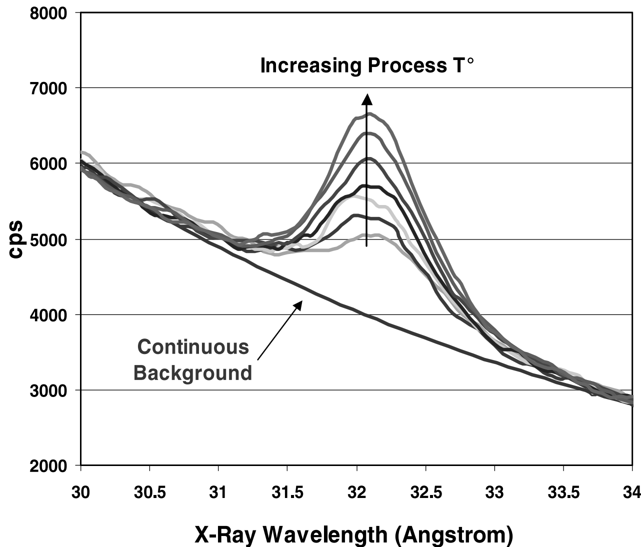


Figure 1. Series of LEXES spectra around the nitrogen K emission from various nanometric oxy-nitride films processed at increasing temperatures. The spectra were acquired in 50 s each using an impact energy of 870 eV. Thousands of counts$\cdot$s^{-1} are obtained, allowing quick and high precision metrology.

Measurements in Small Patterns

A chip consists of many tiny specific areas, repeated hundreds of times at the wafer surface. Each of these areas has sizes typically ranging between one and several hundred microns, *typically tens of microns*.

Throughput

Due to the high productivity standard in semiconductor industry, metrology tools must also demonstrate the capability to test product wafers at high rates. Typically the throughput value ranges between 5 and 20 wafers per hour, sometimes even higher for some real "in-line" applications.

Process Stability

The time of existence of a product can be months or even years. The stability of the metrology tool is defined as the capability to repeat identical measurement on the mid-term and long-term bases (months), as illustrated in Figure 2 for gate oxide process control. Stability values better than 0.5% are typically required.

Process Matching

Manufacturers often fabricate the same product in various factory lines or even in various factory plants. To ensure the uniformity of the products coming from these various sites, it is mandatory that the metrology tools dedicated to the control of the involved process deliver identical responses at the various sites. This capability is called the tool-to-tool matching performance. Matching at better than 0.5% is typically required.

Research and Development of the Chip Process and Characterization Needs

Controlling Composition and Thickness of Thin Structures

To obtain chemical composition and thickness controlled at the sub% precision the process tools have to be calibrated very accurately, by referring to the metrology tool. The latter thus contributes to the elaboration of the process recipes to be transferred to the fabrication lines. Figure 3 illustrates the use of LEXES to optimize the doping level in sub-50-nm ultra shallow junction technology.

In-Depth Profiling

Sometimes the layers are not homogenous and intentionally contain concentration gradients in depth. Ensuring that the species are located at the proper depth is a crucial piece of information for many shallow sample processing techniques. The required depth resolution for this purpose is 1 nm or so.

Lateral Uniformity

The increasing wafer diameter (current standard 300 mm) adds to a previously existing challenge, which is to guarantee that every chip is processed identically whatever its position over the wafer surface. The only real way to check the uniformity of a process is by inserting the processed wafer into a metrology tool having the capability to inspect any point of the surface. Usual mapping schemes involve 5, 9, and up to 49 points distributed over the wafer. Figure 4 shows evidence of how the LEXES can produce uniformity information directly linkable to the chip's electrical properties.

The Various Assets of LEXES as a Semiconductor Metrology Technique

First let me mention that LEXES, like all electron-probe-based techniques, is a compact method that does not require cumbersome or heavy instrumentation. This makes it meet one important basic criterion to be compliant with modern industry.

Its good spatial resolution thanks to the use of electrons for sample excitation is quite a plus. Contrary to X-ray induced techniques (like X-ray photo-electron spectroscopy [XPS] or X-ray fluorescence [XRF]), the LEXES can easily maintain high power and thus high sensitivity and precision in spots as small as 10 μm. This makes it compliant with the examination of the so-called test patterns, that is, some small areas specially designed and distributed over the product wafer surface to serve as reference

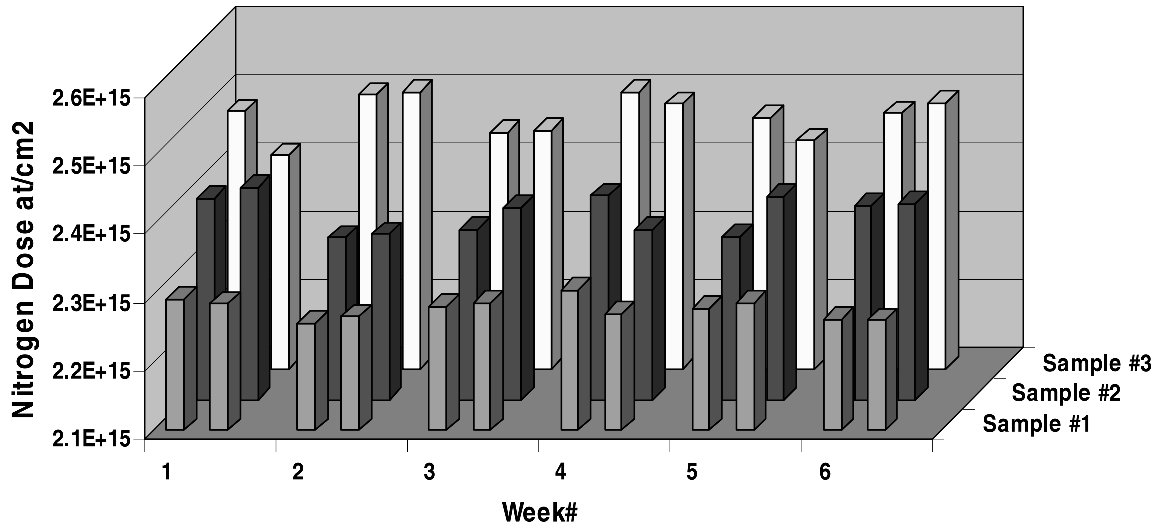


Figure 2. Outcome of a stability test performed over 6 weeks on a shallow probe LEXES tool, measuring twice a week the same three ultrathin gate oxy-nitride samples (2 nm thick). The ability to discriminate between the nitrogen doses injected in each sample was easily maintained all over the period.

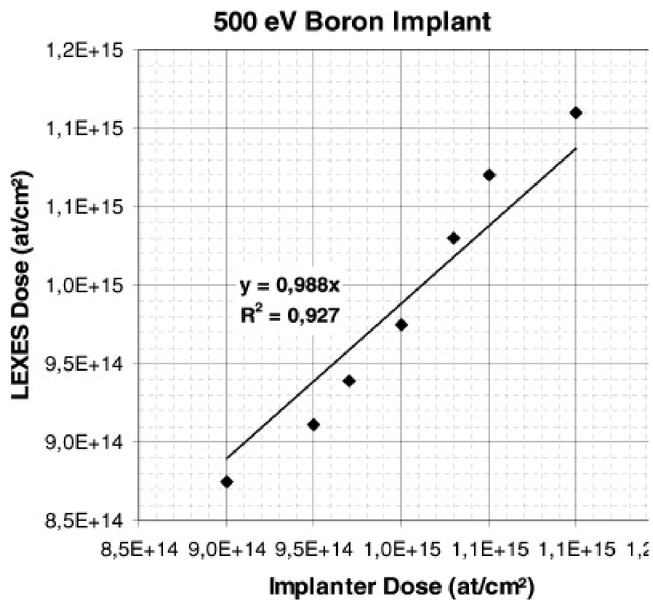


Figure 3. Boron dose measurement with the shallow probe LEXES tool on a series of seven wafers implanted at very low energy (500 eV) with implanter tool nominal values incremented by small amounts (5% and 2% increments). The LEXES response curves evidence some nonlinearity of the implanter tool when such fine-tuning of the implanted dose is carried out.

measurement sites for the metrology tools. This strategy is called near-line or in-line process control.

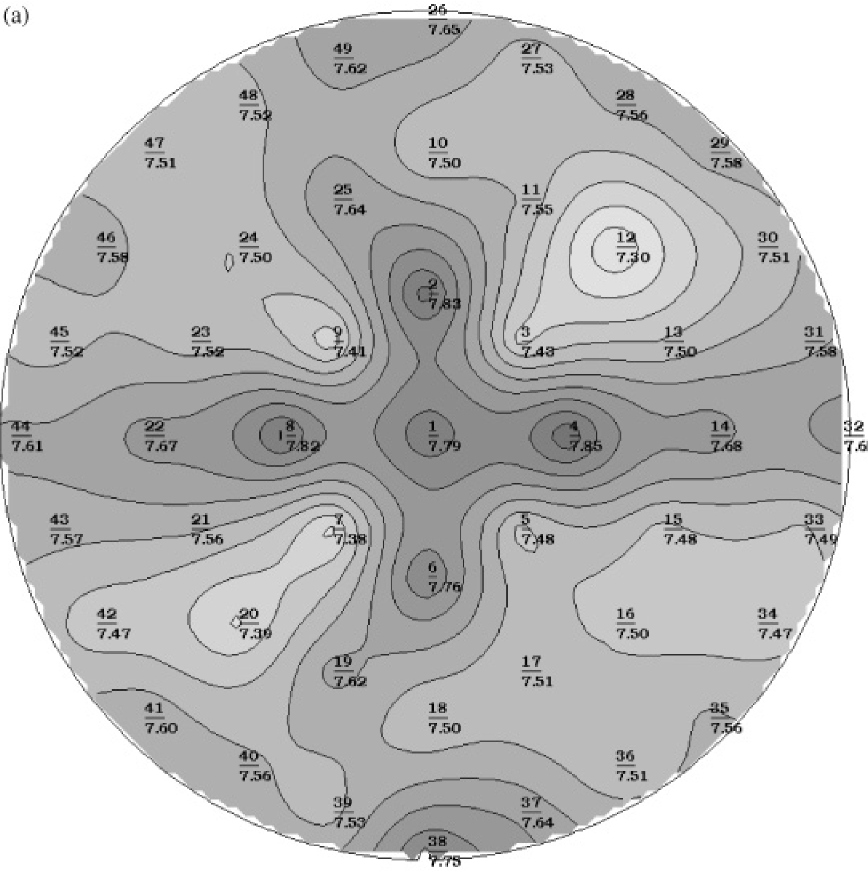
The fact that LEXES provides true elemental analysis is also of high interest, that is, the signal comes from the element itself, and not from some "element-related" information like in those techniques that use sample electrical or optical properties to control the material processing (such as the sheet resistance technique, capacitance voltage, carrier illumination, etc.). Indeed this gives a much more direct insight into the sample and offers quicker process tool troubleshooting.

The deposited power per unit area is also significantly smaller than in the usual electron probe technique like EPMA or Auger due to both lower impact energies and a less focused beam (100 times less current density). So one can inject quite a large amount of electron current (up to tens of microamperes) to obtain sensitivity and reach signal precision down to the 0.1% region.

For research and development applications, when the processes are not mature and undergo variations, an important asset is that even if some significant changes are brought to the material composition, this will not prevent a good interpretation of the emitted LEXES signal. These changes could either be some chemical changes such as variation of the material composition or physical changes affecting the crystalline nature of the matrix, such as annealing, as Figure 5 well illustrates.

The fundamental reason for such robustness is the relatively weak, well-identified, and correctable matrix effects upon the electron-induced X-ray emission, ensuring an excellent linearity of the signal versus nominal sample composition. This is very profitable, for instance, when it is needed to calibrate the doping process of a given species in the sample, like in an ion implantation process, as in Figure 6.

One major benefit from these controlled matrix effects is the ability to reliably compare the sample signal with some reference sample signal through an appropriate and dedicated matrix correction model (Staub, 1998), in order



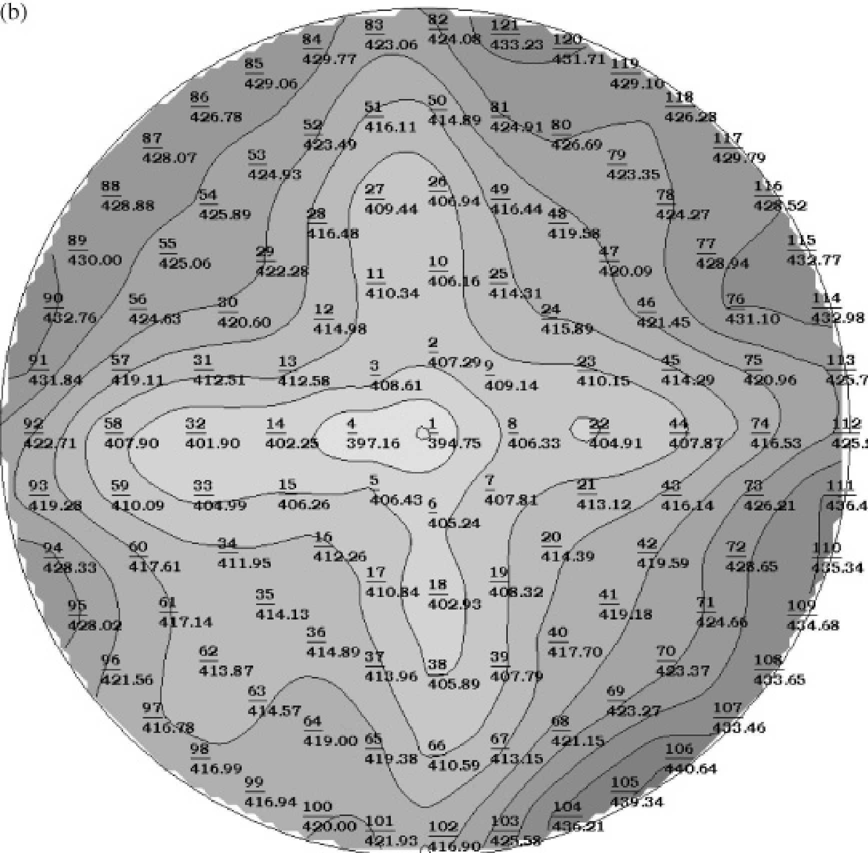


Figure 4. **a:** Forty-nine-point cartography performed on 300 mm wafer, mapping the boron dose measured by LEXES. **b:** The same wafer mapped using the sheet resistance technique, which measures the resistivity of the surface-implanted layer. The double-number labeling of each point in the maps indicates, respectively, the point number (e.g., 1 to 49 for **a**) and the measurement value (in units of E14 at $\cdot$ cm^{-2} for **a**, and some resistivity value for **b**). The wafer rotation was intentionally stepped four times during the implantation process to exacerbate the patterns. As can be seen there exists a nice correlation between the two techniques, highly doped regions corresponding to low resistivity, as expected from theory.

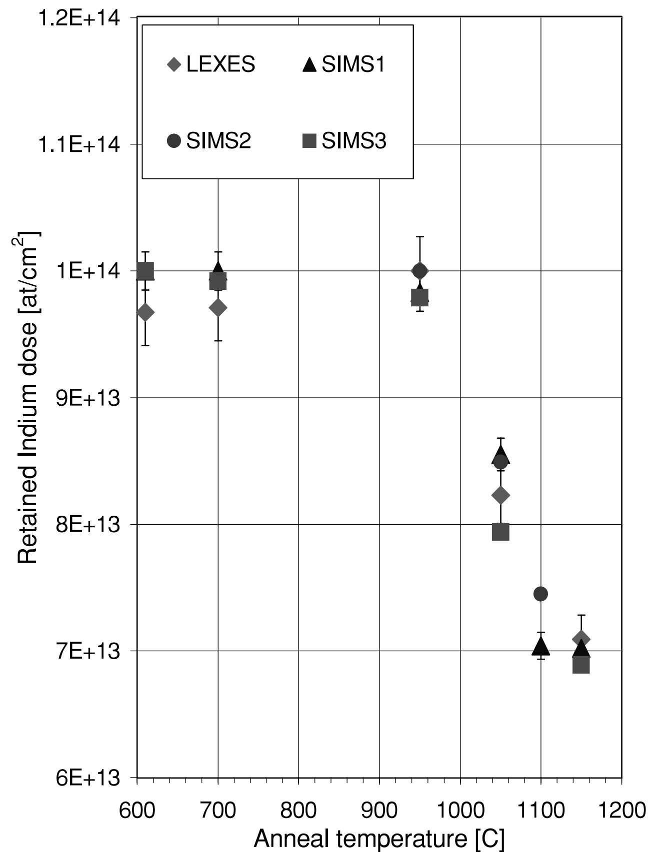


Figure 5. Study of the dose loss occurring during the annealing of an indium-implanted sample. The indium dose retained in the sample, as measured by LEXES and three different SIMS instruments, is plotted against the annealing temperature. The correspondence between the SIMS and the LEXES results is quite good.

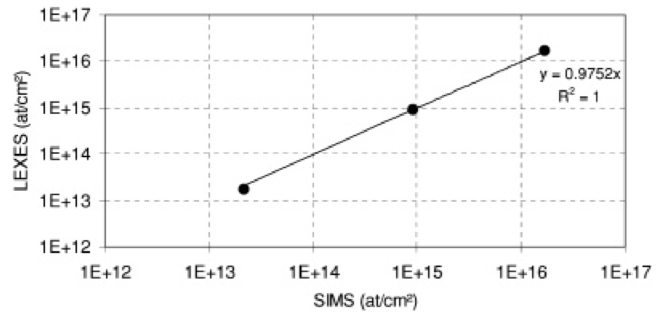


Figure 6. Comparison between SIMS and LEXES arsenic dose measurements from three 100-keV implants prepared by the NIST. Both techniques use the same reference sample (a NIST certified As implanted silicon). As can be seen in the plot, the linear correlation is perfect over this 1000 dynamic dose range (between 2E13 and 2E16 at.$\cdot$cm^{-2}).

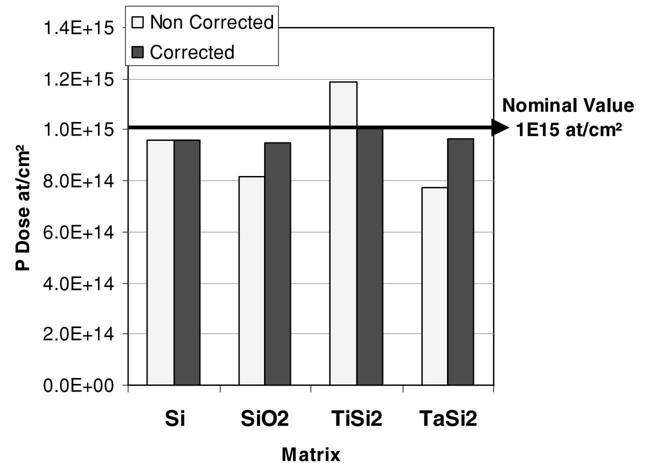


Figure 7. Analysis of four totally different materials (Si, SiO_2, $TiSi_2$, $TaSi_2$) implanted with the same nominal phosphorus ion dose 1E15 at.$\cdot$cm^{-2}. Light color bars indicate the modeled doses assuming the same Si matrix for all samples. This leads to 20% scattering between the determined dose, due to noncorrected matrix effects. The dark bars show the nice improvement brought by modeling correctly the matrix material: The modeled doses differ by less than 2%, despite the big matrix variety.

to produce accurate quantitative measurement, just as in classical EPMA. In many cases, the reference sample can even be very different from the structure of an unknown sample; it just needs to contain the analyzed species in a known and reasonable amount, with a known in-depth structure. Thus pure stochiometric bulk specimens can be used to serve as reference samples to quantify thin films, while maintaining quite decent accuracy. This situation is very rare among other metrology techniques and offers a real comfort to the analyst to start with a new analytical problem. Figure 7 reports how the used matrix effect correction model can ensure the integrity of the LEXES information considering a wide range of usual semiconductor materials.

Another big potentiality of the LEXES comes from the natural in-depth selectivity that is offered by the use of low energy electrons as excitation particles. Indeed, electrons of 1 keV or so lose their energy rapidly as they penetrate the first nanometers in the material, producing distinct changes in their X-ray excitation power. This provides in-depth discrimination capabilities, as two samples with different in-depth structures will generate two different and specific X-ray signal responses as a function of electron impact energy. The capability to probe an in-depth profile by running LEXES in the so-called multi-impact energy mode is largely used in our work, with three different purposes:

1. To ensure an accurate implanted dose evaluation in implantation process metrology.
2. To evaluate the thickness of a homogenous film by combining the modeling means provided by the simulation model IntriX (Staub, 1998) and some reference thin

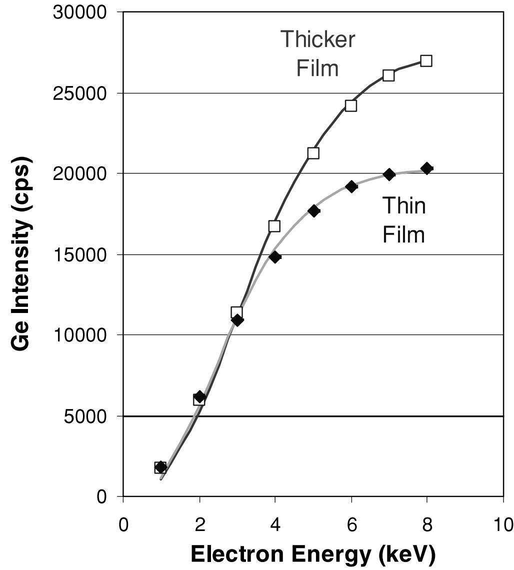


Figure 8. Germanium X-ray intensity as a function of electron impact energy for two SiGe films with thicknesses, respectively, of 100 nm and 80 nm. The X-ray curve shape is a signature of the film thickness. Continuous line curves represent the model fitting.

film material of known thickness. Indeed, the shape of the X-ray signal variation curve when varying electron impact energy is primarily dependent on the film thickness, as illustrated in Figure 8 for a silicon germanium application.

3. To detect in-depth variations inside the processed samples, and thus characterize or make anomaly control of those parameters that govern the profile depth, such as ion implant energy or annealing conditions. The capabilities of LEXES in this respect are well pictured in Figure 9, through comparison with SIMS profiles acquired on various boron-containing "delta layers." Delta layers are ultrathin boron layers deposited onto a silicon surface via thermal evaporation and then buried at various depths inside the samples by growing on their top some silicon caps of various thicknesses.

Finally, LEXES offers the semiconductor engineer the capability to measure with comparable performance almost any species heavier than lithium, and to do so in a crucial range of material thicknesses (from the surface to ~1 μm deep).

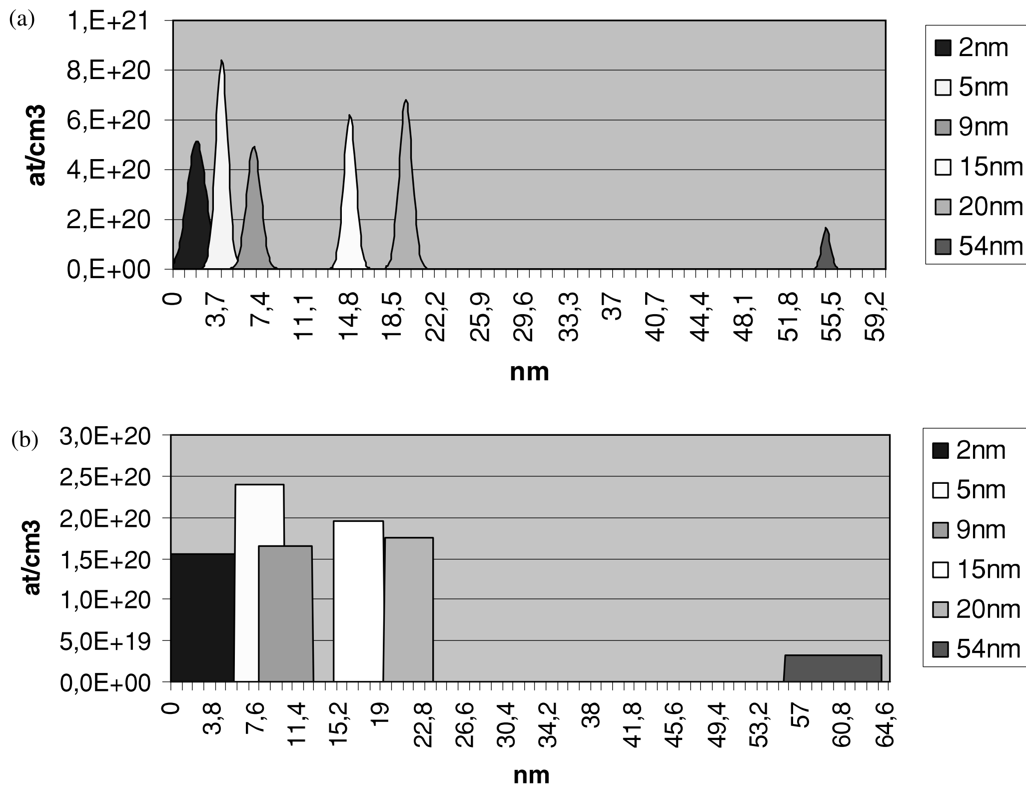


Figure 9. a: A series of ULE SIMS profiles performed on boron delta layers embedded at various depth into a silicon matrix. **b:** Boron in-depth profiles as extracted from some LEXES measurements and modeling on the very same samples. As can be seen, the correspondence is good, indicating that LEXES can accurately locate such ultrathin structures and resolves them at the nanometer scale.

Conclusion

Implementing the low energy electron-induced X-ray emission spectrometry technique as a metrology tool provides several key advantages to characterize and control the processing of many critical and new materials in the semiconductor technology. This technique offers the rare combination of precise and accurate elemental analysis, on small patterns and for ultrathin structures, while maintaining the high productivity that is required in the semiconductor manufacturing environment.

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