Thickness and Composition Measurements of Nanoelectronics Multilayer Thin Films by Energy Dispersive Spectroscopy (EDS)

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Abstract. Scaling is one of the main aspects to continuously improve the performance of nanoelectronic devices. This leads to increasingly more complex stacks including new materials; for example SiO₂ is being replaced by high-k materials for gate dielectric applications and metal gates are introduced in place of poly-Si. Moreover, a change in the type of substrate is also considered; Ge and III/V materials are intensively investigated as possible candidates for Si substrate replacement. The introduction of these new materials together with the device downscaling has drastically increased the demand for both composition and thickness metrology methods. Various established techniques providing composition analysis exist such as X-Ray Photoelectron Spectroscopy (XPS) and Rutherford Backscattering Spectrometry (RBS), however, they usually require a large area for the analysis, complex instrumentation and can be time consuming. In this paper, Energy Dispersive Spectroscopy (EDS) coupled to Scanning Electron Microscopy (SEM) is used to determine both composition and thickness of simple and more complex multilayered thin films. The use of EDS for thickness measurements in small scale areas is also investigated.

1. Introduction
The analysis of thin films has become very important nowadays with the downscaling of nanoelectronic devices. In this context, we see in the most recent technology generation for complementary metal-oxide semiconductor (CMOS) applications a tendency to replace the Si substrate by Ge or III/V materials to increase the carrier mobility. This is also accompanied by the replacement in the stack of both SiO₂ and poly-Si by high-k materials as gate dielectrics and by metal gates, respectively. This has lead to a strong increase in the number of elements used in the semiconductor industry, a number that has been multiplied by a factor of around 6 during the last 20 years as shown in Figure 1. Controlling or knowing the real thickness and composition of the films in these multilayered stacks is crucial for electronic research and development. To determine both thickness and composition of thin films, several techniques could be used, such as X-Ray Photoelectron Spectroscopy (XPS), Rutherford Backscattering Spectrometry (RBS) and Medium Energy Ion Scattering (MEIS). These techniques however require large and expensive instrumentations, are usually time consuming and destructive and do not have a small analysis spot. On the other hand, Energy Dispersive Spectroscopy (EDS) coupled to Scanning Electron Microscopy
(SEM) has the potential for fast analysis on small scale areas. In our previous work [1], we used mainly EDS to determine the thickness of both single and multi-layer systems. Very good correlation with other analytical methods was found down to a layer thickness of 0.5nm. In this paper, we show how EDS can be used to determine at the same time with a high accuracy both thickness and composition of complex multi-layer systems with a wide range of elemental composition. Moreover, we will show that EDS when coupled with SEM enables the thickness determination of advanced thin films in small areas.

2. Experimental

The EDS experiments were performed with two Oxford Instruments INCA Energy 350 Microanalysis systems including a 30 mm² INCA PentaFET-x3 Si(Li) detector. The resolution of these systems is better than 133eV at Mn Kα for count rates up to 4000. The EDS systems are mounted on two different SEM microscopes (FEI XL30 and FEI Nova200). The obtained EDS data were treated using both INCAEnergy and ThinFilmID software from Oxford Instruments. Calibration standards were measured in the same conditions as the samples under investigation.

3. Results

3.1. EDS for thin film analysis

The basis of the EDS technique is to measure the X-ray line intensities generated from the sample after irradiation by an electron gun. It is well known that these X-rays are generated not only in the top outer part of the sample but are collected from a volume of a few cubic microns of material [2]. The ThinFilmID software is taking advantage of this ‘effect’ of penetration of the beam in the analyzed sample in order to retrieve more information about the disposition of the elements. For doing this, the collected EDS spectrum by itself is not sufficient, but a description of the sample is also required. The X-ray intensities predicted from the theoretical model of the structure are then compared with the intensities obtained from the X-ray spectra. By using an iterative procedure, the thickness and composition at which the model matches the experimental data are calculated for each layer in the model. The calculation is calibrated using the X-ray intensities from bulk standards.

As an example, ThinFilmID was used to determine the thickness of a Pt metallisation layer deposited on SiO₂ on Si substrate. EDS spectra were recorded using two accelerating voltages of 10 and 20 kV. As shown in Figure 2, both SiKα and PtMα lines are observed under both conditions, the ratio of these lines being different due to the difference in beam penetration. Using a simple model where only the Pt layer thickness is allow to vary, the results reported in Table 1 were obtained. We could see that the thickness calculated from both accelerating voltages is in accordance with the nominal value. To confirm the validity of the ThinFilmID technique, the Pt metallisation layer thickness was also measured with RBS (see Table 1). The obtained result confirms the accuracy of the ThinFilmID procedure.

Figure 1. Number of elements used in semiconductor industry (source: sematech / intel).
3.2. EDS analysis of multilayered systems
The use of ThinFilmID has been applied to more complex systems, i.e. multilayered systems where both thickness and composition have to be determined at the same time. Figure 3 describes the stack that has been measured. It consists of an intermediary SiO₂ layer on a Si substrate where the thickness has to be determined. The top layer consists of TiN where both composition and thickness should be measured. Additionally to that, Al and Cl impurities were intentionally introduced in the TiN layer. Various impurity levels were used; this leads to the definition of three series of samples, i.e. A, B and C with no, low and medium Al contents, each series having also three different Cl contents, as indicated in Figure 3. In this context, ThinFilmID has been applied to determine at the same time the thickness of both SiO₂ and TiN layers together with the composition (including impurity levels) of the TiN layer.

The results obtained from ThinFilmID are presented in Figure 4. It shows that the SiO₂ thickness is quite similar for every sample of each series and very close to the nominal values (20nm). On the other hand, we could see more variations in the TiN layer thickness. The three different Cl levels incorporated in the samples of series A (no Al) are clearly identified, i.e. a low, medium and high
concentration is measured. A minimum value of 1 at.% Cl was found. In series B, a low amount of Al is measured, i.e. between 0.5 and 1 at.%, and the three Cl levels are again determined. Finally, similar results are observed for series C, i.e. for the samples with a medium Al content. To evaluate the accuracy of the EDS results, independent RBS analyses were performed on the same series. A relatively good correlation between the values obtained from both methods is found, as displayed in Figure 5.

Figure 4. (a) TiN and SiO₂ thicknesses and (b) Aluminium and Chlorine concentrations determined from ThinFilmID.

Figure 5. EDS and RBS correlation for (a) Chlorine and (b) Titanium concentrations.

3.3. Thickness measurements of patterned thin film structures
The ability of EDS to measure the thickness and composition of thin films has been demonstrated before. However, as indicated in the introduction, the size of the devices is not only decreasing in thickness but also in lateral dimension. It is therefore important for small patterned structures to evaluate the lateral size limitation for an accurate thickness measurement. To test the practical application of ThinFilmID to this localised measurement, a test wafer has been used (Figure 6). The wafer consists of nominally 128nm thick SiGe (nominally 24 at.% Ge) structures on a Si substrate in a range of sizes, including square structures of length 10µm, 5µm, 1µm, 500nm, 300nm, 200nm and 180nm. The SiGe structures are surrounded by Si₃N₄/SiO₂ areas.
EDS spectra were collected from the centre of each square SiGe structure. The thicknesses of these structures as measured by ThinFilmID are plotted against the lengths (lateral dimensions) of the squares in Figure 7(a). The full line presents the results obtained with the nominal value of 24at.% Ge in the model. It shows that for the structures of 10µm, 5µm and 500nm an accurate thickness measurement of ~128nm is obtained. These values are in good agreement with the TEM results, as shown by the example of the 5µm SiGe structure displayed in Figure 7(b). The error on the calculated thicknesses for smaller structures is however larger, i.e. up to 10% for the 300nm and even more for the 200nm and 180nm square structures. This error is due to the generation of X-rays from the Si₃N₄/SiO₂ stack adjacent to the structure. The proportion of this surrounded area in the EDS spectrum is increasing with reducing structure sizes and thus explains the increase of the error observed on the thickness value.

The measured thickness for the 1µm structure is however far from the expected value and could not be explained by the structure size limitation. To overcome that problem, Atom Probe (AP) analysis of this specific size has been performed. In AP, a strong electric field induced by applying a bias of several kilovolts is needed to allow the ejection of atoms from the sample surface; however, to achieve
this high electric field the sample has to be manufactured in a tip-shape structure. Figure 8(a) shows the tip prepared from the 1µm square SiGe structure using Focused Ion Beam (FIB) milling, the Pt cap layer is used for sample preparation. The 3D AP reconstruction of the tip is displayed in Figure 8(b). The various components of the tip are clearly observed, i.e. Pt cap layer, SiGe structure and Si substrate. Because of Pt incorporation in the first nm of the SiGe film during FIB preparation (indicated by the intermediate layer), the thickness of the SiGe layer could not be determined precisely. However, the SiGe composition could be easily determined. A value of 21at.% Ge could be measured, which is lower than the nominal 24at.% value. Previous work has demonstrated that AP is quantitative in that range of SiGe concentrations [3]. This difference of concentration could of course be the source of error in the EDS thickness determination. A new ThinFilmID calculation was therefore made using 21at.% Ge in the model for the 1µm square structure; the results are represented by the dotted line in Figure 7(a). A SiGe thickness of around 132nm is found which is in agreement with the TEM results.

4. Conclusions
In this work, we show that using proper modelling of the investigated systems, EDS can measure with high accuracy both thickness and composition of multilayered stacks. This is demonstrated by the good correlation existing between the EDS results and independent measurements by RBS, TEM and Atom Probe. It was also shown that the thickness of patterned structures can be measured accurately using ThinFilmID down to lateral sizes of at least 500nm. This is significantly smaller than the 0.5-1mm window sizes often required for other techniques.

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