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1. Is the deliverable in accordance with						
(i) the Description of Work?	☒ Yes ☐ No		☐ M ☐ m ☐ a	☒ Yes ☐ No		☐ M ☐ m ☐ a
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(iii) that needs further work by the partners responsible for the deliverable?	☐ Yes ☒ No		☐ M ☐ m ☐ a	☐ Yes ☒ No		☐ M ☐ m ☐ a

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Abstract

The project partner IISB performed additional measurements on prepared oxidized Al and Cu bond pads, and metal stacks samples. The objective was to acquire statistical measures such as repeatability and accuracy of the EDS measurement using the new detector type to gain a solid understanding of its potential use as an analytical tool and its usability at an end user site, i.e. in a semiconductor production process environment. The main focus of this project period was to cross check EDS results with alternative analysis techniques and to work out the reliability and limitations of the individual methods. Also the analysis speed and sample preparation issues have been evaluated.

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1. Introduction

The aim within this project period is the development of a routine analysis scheme for the fast analysis of thin functional layers enabling reliable monitoring and control of production processes. Therefore, the plan was to examine the samples produced by the project partner with alternative measurement techniques. A thorough comparison of the results from different methods was conducted, and the sensitivity and differences of the respective methods were discussed. The specific errors and variations of the methods are discussed in order to work out a reliable standard analysis procedure for the thickness determination of thin layers by EDS analysis.

D14.1.3 is the related deliverable. It includes the cross check of measurement data by various analysis methods including direct layer thickness determination results.

2. State of the Art

The reference samples are not exactly state of the art dielectric layers on silicon wafers. They were specifically tailored for the needs of the NaREA project. We still use the first and second set of samples supplied by IFX. The production type samples will be included in the next step.

- The first reference series is CuO_2 on Cu on SiO_2 on Si substrate
- The second reference series is Al_2O_3 on Al on SiO_2 on Si substrate
- The layer thicknesses are 2, 10, 20, 30, 40, 50 nm as pre evaluated by IFX after fabrication.

At present the production process control measurements at the end user site (IFX) is performed ex-line by beam profile reflectometry (BPR). This technique requires a set of optical constants for a particular material for the given wavelength of the device. The layer thickness will be determined by a calculation from the fit of the peak profile measured to a theoretical model.

Alternatively the layer thicknesses are measured with XPS or Raman spectroscopy which is time consuming, taking a measurement time of around 45 min per measured point and additional time for the evaluation of the measured spectra, plus the layer thickness calculation. In total the time taken is around an hour per single measurement.

With the conventional EDS detectors it was so far not possible or trusted that the measurement of thin layers would be possible with high enough reliability, even if the layer thickness was determined from one spectrum with single acceleration voltage, like it is done by the Oxford system. The common systems require the measurement of the layers with at least two acceleration voltages, which would at a minimum double the time for a single measurement.

The study of the copper oxide is especially difficult since this layer material can vary in oxygen content. The composition must be determined either simultaneously or before the thickness evaluation. This is not possible e.g. by the ellipsometry. EDS can function for both. However, the quantitative ratio between the metal atoms and the oxygen in the case of the copper oxide is reduced from CuO to Cu2O. This lowers quantitatively the absolute fraction of oxygen in the Cu2O layer compared to CuO. It was to be demonstrated where the limitation for the EDS method lies and if it is still possible to measure the Cu2O layer thicknesses.

During the NaREA project it was possible to show that the EDS technique utilizing the new single chip SDD detector with the larger solid angle, results in a good signal quality at short measurement times. The related software for the simulation of the EDS spectra (e.g. the

models applied), the quantification process and the calculation of the layer thickness was evaluated in depth.

3. Results and Analysis

The samples mentioned above have been further analyzed with EDS including repetitive measurement to obtain statistical data such as accuracy (absolute and relative) and repeatability. This data was then compared with measurements from alternative analysis techniques.

The methods of choice for comparison purpose were ellipsometry, x-ray reflectivity (XRR), beam profile reflectometry (BPR), and direct layer thickness determination from cross sections by scanning electron microscopy.

The NaREA project experienced support from SP1 for the ellipsometry measurements in the frame of cross cut activities, such that also a deeper evaluation of the performance, potential and time requirements of techniques will be possible within SP1 and NaREA.

The various analysis methods determine layer thickness out of different parameters and material depending physical properties. Whereas the ellipsometry is sensitive to the optical properties (dielectric function) of a layer and surface quality, the EDS method mainly is determined by the chemical composition and x-ray density. The XRR is basically sensitive to the electron density distribution in a sample and to structural issues like surface roughness or roughness of interfaces. The direct methods selected are SEM or transmission electron microscopy (TEM). The SEM imaging of the layers in cross section was selected to avoid increased work load which is necessary for a TEM film preparation.

For the measurements of the Al2O3 layers on Al on silicon the optimum acceleration voltage was determined below 3kV. This becomes obvious from the k-factor sensitivity plot of oxygen K-line (which is used for the quantification), expressed as the intensity ratio of the oxygen/copper lines used, as a function of layer thickness. The plot shows intensity ratios calculated for three different acceleration voltages at 3kV and below (see Fig. 1 off the annex).

The slope of the graphs develops larger with lower accelerating voltage, thus indicating that the sensitivity of the method using a light element line such as Oxygen K-line could be improved by lowering the accelerating voltage below 3kV. The simulation by the thin film ID software (TFID) supplied by the partner Oxford Instruments, does come to the same result and proposes a low accelerating voltage.

However, the quantification of the spectra with the TFID below 3kV is judged invalid and is not possible with the current set of profiles underlying the software in its present state. This is a limitation which cannot be circumvented up to now, but does not mean that the determination of layer thicknesses from those layers is impossible. The limitation to acceleration voltages above 3kV makes sense for most applications due to the fact that mass absorption coefficients or k-factors do not exist below 3kV. Respective models for the calculation of absorption and fluorescence effects are, mainly for historical reasons, not available for primary beam energies below 3kV, and in turn calculations would be wrong.

Here basic scientific work is required to improve the models for quantification and determination of k factors for low acceleration voltages. The EDS analyses relying on the oxygen line were performed with the minimum primary beam energy of 3kV. This affects probably the accuracy of the thickness determination of layers where the absolute oxygen content is quite low like in the case of Cu2O. The conditions are in those cases not optimal yet. Table 1 shows a summary of results obtained from EDS and comparative measurements in the case of the Al2O3 layers. It can be seen that results from cross section investigation as a direct measure do compare nicely with the results from EDS measurements. In the case of

ellipsometry study it is obvious that the measurement with one angle is not suitable and at least three angular measurements are necessary which is most time consuming. The data for the process thickness targeted by IFX (process, BPR), differs slightly from the absolute values attained by the SEM cross section imaging. The XRR failed completely for the determination of layer thickness. The XRR spectra did not exhibit any fringes. This is most often the case if the interfaces and the layer surface is rough that no constructive interference can occur. The results and conclusions were delivered and discussed with the consortium partners during a project meeting.

4. Summary and Conclusion

We evaluated the performance of the new detector for layers that are suitable for EDS measurements using the light element line only. The results were compared to results obtained with alternative measurement techniques. In conclusion it can be stated that the EDS measurements lead to a value for layer thickness of the Al2O3 layers on Al substrate within an error of +/- 1-2nm compared to results from direct layer thickness determination with regard to the value obtained from a direct measure which is very satisfactory. Even more so if one considers the time and labor effort needed to attain a value from a single point, which differs greatly amongst methods.

The pure counting time for an EDS measurement is 60sec even for low Oxygen concentrations. Adding the time required for the quantification optimization and calculation of the thickness the total analysis time would lead to approx. 2 min. For the same measurement using ellipsometry, three angular measurements of about 30-45min are necessary. A similar time period would need to be added for spectrum simulation and thickness value calculation. The BPR measurements did not show the same accuracy compared to the reference value from cross sections. One of the comparative techniques (XRR) failed completely.

Annex (see text for explanation)

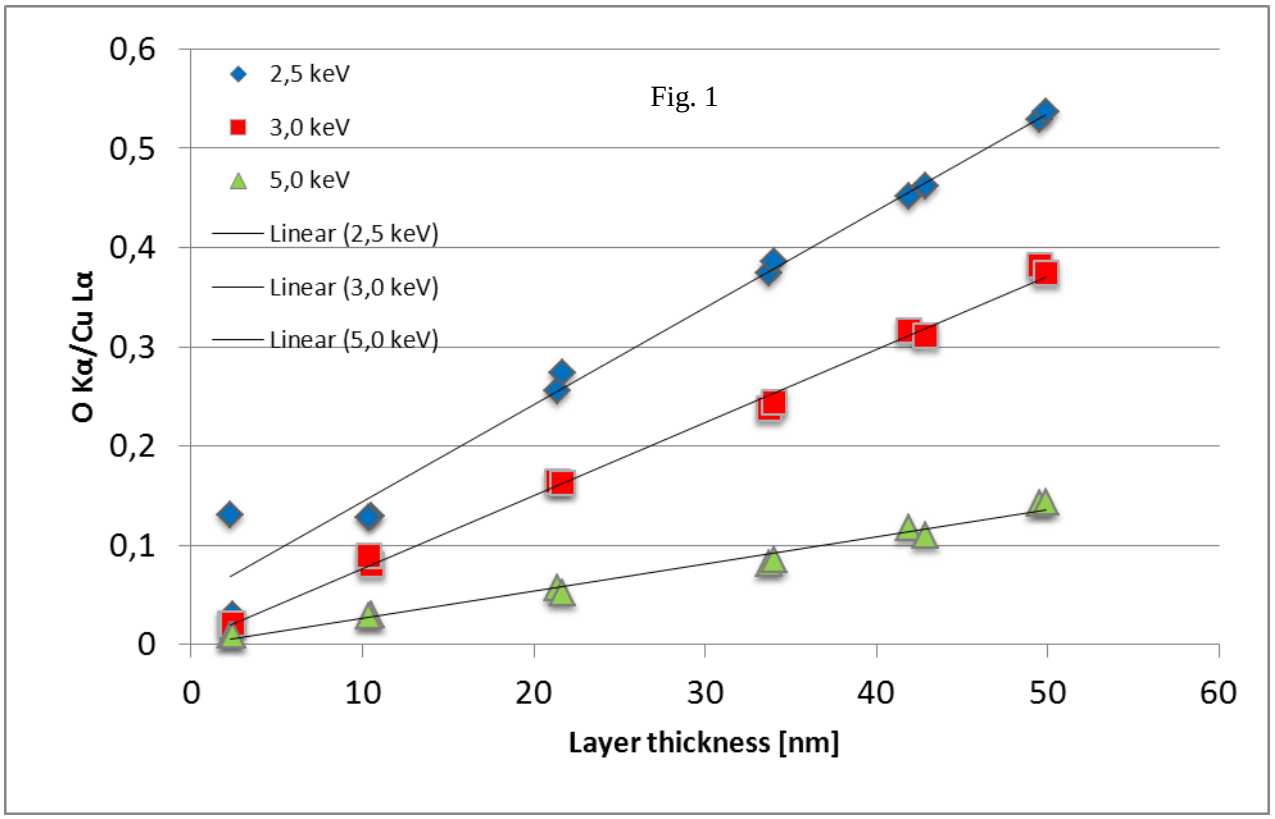

Fig. 1

Table 1

	IFX Process	IFX BPR	Ellipsometry one angle, AOI 1Pt	Ellipsometry 3 angles, AOI 1Pt	EDS	SEM
Specimen #	**Thickness (Al2O3 on Si)**	**Thickness [nm]**	**Thickness 1Pt [nm]**	**Thickness [nm]**	**Thickness [nm]**	**MW [nm]**
6	-	2,26	4,65	3,83	3,09	5,22
7	10,18	10,52	13,09	10,21	10,15	10,19
8	20,52	21,35	23,37	19,64	18,61	18,92
9	30,25	33,76	32,17	29,12	26,86	28,75
10	40,18	41,88	42,34	38,02	40,47	39,31
11	50,75	49,58	52,17	46,90	45,93	47,98
20	-	2,41	4,64	-	2,99	
21	10,18	10,41	12,77	-	9,97	
22	20,52	21,7	22,35	-	18,41	
23	30,25	34,04	31,84	-	28,91	
24	40,18	42,91	42,35	-	37,16	
25	50,75	49,91	51,99	-	44,32	