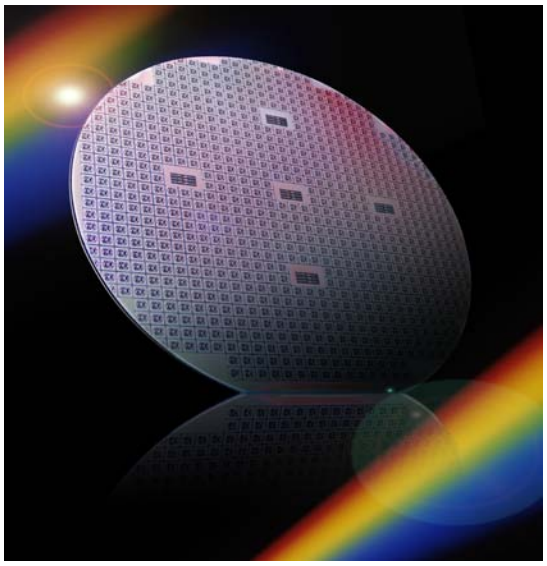
EDXRF APPLICATION NOTE
THIN FILMS ON SILICON

#1054

SCOPE



This App Note demonstrates the capabilities of the Rigaku NEX CG EDXRF analyzer for the measurement of thin film thickness (Tox) applied to semiconductor wafers. Simple 2-point empirical calibrations are used for SiO2, AlSi, Ti, TiN, Pt, AlCu, and BPSG single layer films on silicon substrates for use in quality control in the semiconductor industry.

BACKGROUND

Process control in semiconductor manufacturing is critical. Tight tolerances leave little margin for error in the processing of semiconductor materials. Film thickness and concentration integrities are vital to optimal device performance. Semiconductor processing involves the sequential deposition of multiple layers to complete the device. Any errors during the construction of these devices can lead to undesirable effects in future processing and lead to device failure or degradation.

INSTRUMENTATION

Model: Rigaku NEX CG

Excitation: Indirect with polarization

X-ray tube: 50 W Pd-anode

Detector: High performance SDD

Sample Type: Flat silicon wafer samples

Environment: Helium purge*

Options: Flat window ring

Analysis Times: Monitor wafer samples 100 sec per sample

* NEX CG offers both helium purge and vacuum in the same instrument. Use of helium or vacuum yields the same sensitivity and performance. Vacuum allows for analysis when use of helium is not desired, or when helium is not readily available.

SAMPLE PREPARATION

Various PVD and CVD films were used for measurement. Samples were analyzed with no additional sample preparation. The automatic sample changer (ASC) apparatus was removed from the analyzer and samples were manually analyzed using the flat window ring. Samples between 25-31mm diameter radius can be measured using the 15-position ASC, and samples with diameter 25-39mm can be measured using the 10-position ASC. A maximum 350mm diameter wafer or sample can be placed in the sample chamber for analysis.

CALIBRATION and MEASUREMENT

All single layer films were deposited on silicon monitor wafers with 1000 Å of SiO_2 (used as an adhesion layer). To demonstrate the performance of NEX CG a series of 6 different 2-point calibrations were developed for the thickness of the films AlCu, AlSi, Pt, Ti, and TiN. BPSG composition was determined by measuring P, and the B:P ratio was constant at 1:1. In each calibration a 1000 Å SiO_2 film deposited on silicon was used as the zero point or blank sample. The second point in each calibration was obtained from the calibration standard of interest. A 1-point calibration was developed for the SiO_2 film. Single point or two point calibrations can be used for thin film thickness analysis since the X-ray response is linear at low thickness ranges. As thickness increases the XRF response is curved. For example, the AlSi layer thickness is curved over the range 0-10000 Å, and to fully characterize the entire range at least four calibrations are required.

RECOVERY

Following calibration, all calibration standards were analyzed to demonstrate valid recovery. Results are listed below:

Tox Data		Units: Å	
Sample Film	Standard Value	Average Value*	% Difference
AlCu	9581.6	9539.5	0.4%
AlSi	11689.8	11845.1	-1.3%
Pt	990.3	982.4	0.8%
Ti	1004.8	1004.9	0.0%
TiN	997.6	993.2	0.4%
SiO_2	1000.0	1025.5	-2.6%

** Average value reflects the calculated value from the calibration.*

P Concentration Data		Units: Mass %	
Sample Film	Standard Value	Average Value*	% Difference
BPSG**	4.0	4.1	-2.5%

*** The ratio of B to P is at a fixed ratio of 1:1*

REPEATABILITY

Representative samples were analyzed to determine measurement repeatability (precision). Ten repeat analyses were performed in a static position using a count time of 100 sec per analysis condition.

Tox Data		Units: Å	
Sample Film	Standard Value*	Average Value	Standard Deviation
AlCu	10087.8	10185.3	29.0
AlSi	8275	8279.2	48.9
Pt	1000	1058.3	2.1
Ti	1000	986.0	1.9
TiN	1005	1006.2	2.3

** Nominal values supplied with the samples.*

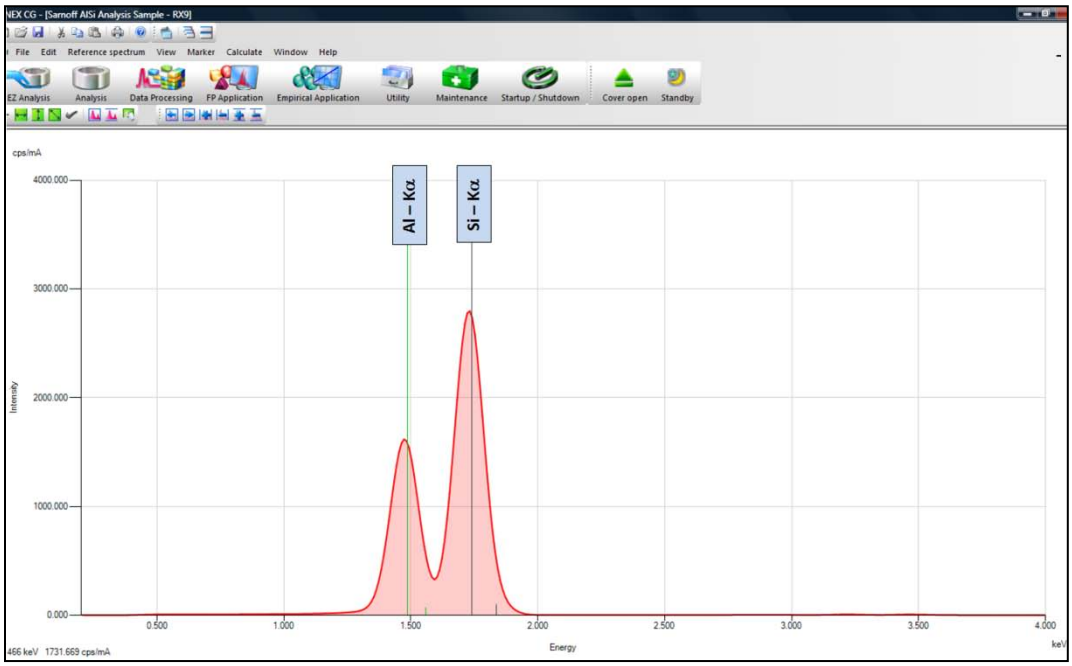
P Concentration Data		Units: Mass %	
Sample Film	Standard Value*	Average Value	Standard Deviation
BPSG**	4	4.2	0.1

*** The ratio of B to P is at a fixed ratio of 1:1*

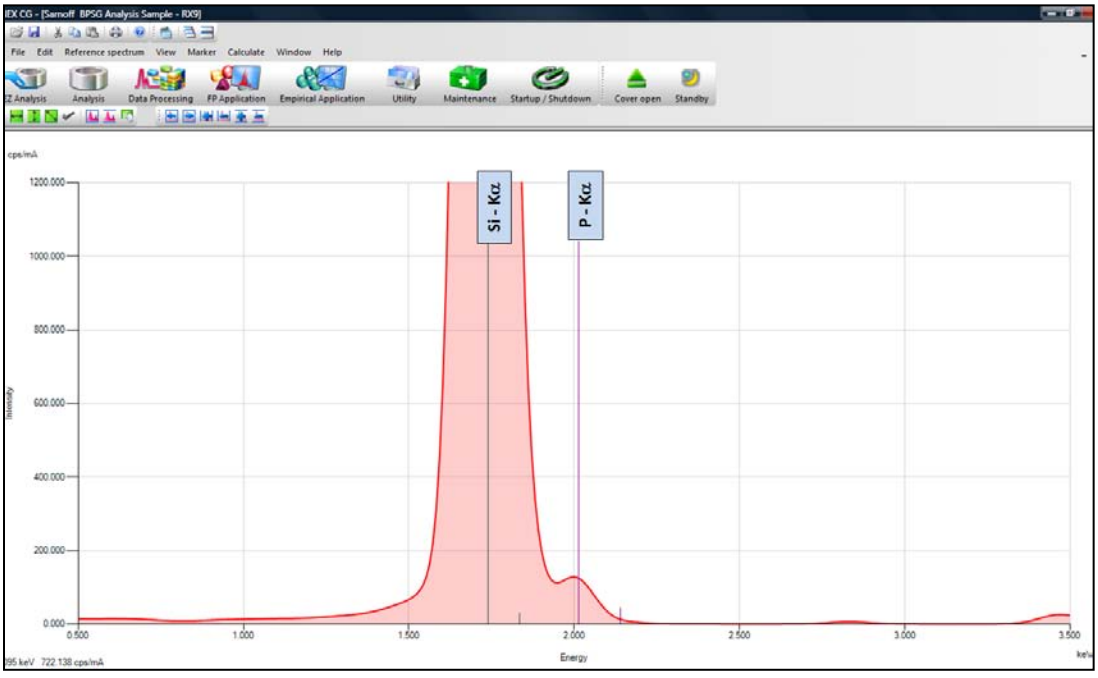
Applied Rigaku Technologies, Inc. ▪ 9825 Spectrum Drive, Bldg. 4, Suite 475 ▪ Austin, TX 78717 USA
www.RigakuEDXRF.com ▪ info@RigakuEDXRF.com ▪ (+1) 512-225-1796

QUALITATIVE ANALYSIS

Excellent peak resolution is observed for each element. Example spectra using the RX9 polarization target are provided below to demonstrate superior light element measurement.



AlSi film using RX9 Polarization Target



BPSG film on RX9 Secondary Target

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DISCUSSION

Analyses were performed in a helium purge environment, which allows for optimum sensitivity measuring the low energy light elements (Na-Cl). Single measurement condition applications were utilized for the analysis of monitor wafer samples. A 100 sec count time was used for each application consisting of a single measurement condition. Complete analysis of patterned wafer samples required three measurement conditions, each with a 100 sec count time. The total count time for patterned wafer analysis was 300 sec.

The composition of the multi-element thin films studied for this report were constant, except for the BPSG film. The P concentration was determined with fixed BPSG film thickness. The NEX CG thin film FP program is a powerful and versatile package that allows for measuring thin film composition and thickness simultaneously, as well as the ability to measure multi-layer films. This gives the operator a valuable tool for many types of thin film applications, as well as bulk analysis of liquids, solids and powders.

The NEX CG offers the value and flexibility to be used as a monitoring tool throughout the semiconductor process cycle. The NEX CG can provide routine monitoring for dopant concentration (CVD and Diffusion), thickness uniformity (CMP), etch rate (Wet and Dry Etch), and absolute thickness (Metallization) processes. The NEX CG can also be used to identify trace contaminants that could potentially lead to device failure.

CONCLUSION

The NEX CG offers analysts a simple yet powerful and versatile system for indentifying and quantifying elemental composition. The results of this study indicate the Rigaku NEX CG EDXRF analyzer with indirect excitation and polarization is an excellent tool for screening and quantifying thickness, concentration, and trace contaminants for semiconductor processing materials. The NEX CG can reliably measure elements from Na to U and this versatility will allow the NEX CG to adapt as existing processes are refined and new processes are introduced.

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