Thin Film Analysis with the ORION® PLUS Spectra

Author: Larry Scipioni
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Application

This application note discusses the measurement of film thickness for films grown by Atomic Layer Deposition (ALD) or related techniques. This task is particularly important for qualifying liners or high-k dielectric layers which are critically controlled to be very thin.

ORION® PLUS Spectra Capabilities

A novel ion spectroscopy method is provided that takes advantage of the extreme surface sensitivity in helium ion scattering. Energy proportional backscattered particle detection makes it possible to obtain spectra in the ORION® PLUS tool. Simulation software allows fitting the data to models for deriving film thickness.

Background

The creation of very thin films is key to enabling many device properties. In this note we focus on the deposition of zirconium oxide as an example of a process development or monitoring task. Gate oxide thickness for a high-k/metal gate is required to be on the order of 1 nm or less. Therefore the technique used to measure the film needs monolayer sensitivity in the growth direction. If the film is patterned or inhomogeneous a spatially resolved technique is also required.

Challenge

The measurement of such thin films can only be accomplished by certain techniques. One such method with the required surface sensitivity is Rutherford Backscattered Spectroscopy (RBS). Measurements with RBS reveal the number of target atoms per unit area, with the ability to detect single atomic layers. This technique uses high energy (usually 1-3MeV) helium ions as the probe. Carrying out such a measurement requires a dedicated station at a high energy ion accelerator, however, requiring the user to utilize a special facility. The lateral resolution of RBS is very low as well, typically about 1 mm. (Specialized micro-RBS systems can achieve resolution around 2 µm.) Also, such systems do not offer any imaging capability for selecting an analysis region. These two factors present a barrier for performing analyses on patterned films. While there are standalone tools that can perform analogous techniques such as medium energy ion scattering, they remain as fairly expensive, single use systems.

ORION® PLUS Spectra Solution

The ORION® PLUS Spectra provides helium backscattering onto an already highly capable microscope, allowing elemental analysis on top of record-breaking imaging resolution performance. A specially modified silicon drift detector is provided in a sample-facing port that allows the acquisition of spectra either in a spot mode or while imaging. The spectrum collected is a total of the entire scanned area (no elemental mapping). The user chooses the region of interest via the image. The minimum analyzable volume depends on the sample material and geometry and is not yet fully characterized for this novel technology, but it is known that the sampling volume is smaller than, for example, in EDS. Three initial applications are identified for our technology: small particle analysis, elemental profiling across a surface, and thin film analysis. The latter of these three is the topic of this note.

We describe the thin film measurement technique through an example. A set of thin films of ZrO_x on silicon with native oxide was created by ALD. The number of cycles of ALD was varied for the samples in the set. A spectrum from each sample was captured with the ORION® PLUS Spectra; these are shown in Figure 1 at the right side. The primary beam energy was 35 keV, which appears as the maximum ordinate value on the plot. With a helium ion beam current of 20 pA, spectra are captured in about 1 minute each with a 12.2 eV channel width. The analyzed area varied, but for the 30-cycle film we analyze here, the irradiated area was 5 µm × 5 µm. Since the resolution of the detector is about 4 keV for helium particles, the data is filtered by means of a running average in a 300 eV wide window. This manages noise without sacrificing data integrity. The peaks seen at higher energy correspond to the zirconium, while the rising edges at lower energy are the backscatter signals from the silicon and oxygen. Two facts can immediately be discerned from the smallest two zirconium peaks: this technique has monolayer detection capability, and the sensitivity is high enough to easily distinguish the 1-cycle from the 3-cycle ALD sample. A set of RBS data were also gathered from these films, and it was found that the intensity of the zirconium peaks captured in the ORION® PLUS correlated consistently with the RBS data. This is shown in Figure 2. There is an average scale factor of 0.79 ± 4 % between the sets, and the consistency of this offset makes it possible to calibrate very well for a given application. This provides a strong reference to an established technique.

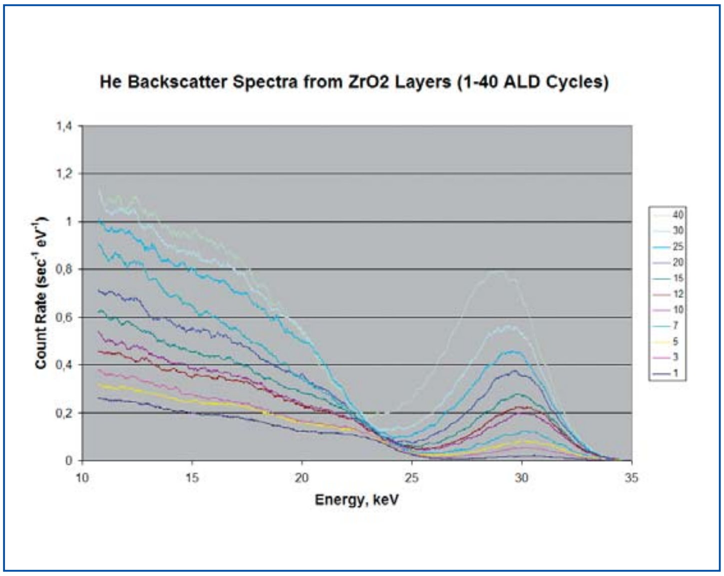

Figure 1: Helium backscatter spectra from a set of ZrO_x films on silicon. The legend indicates the number of ALD cycles used to grow the various films. Samples provided courtesy of Dr. Steffen Teichert, Qimonda (Dresden, Germany).

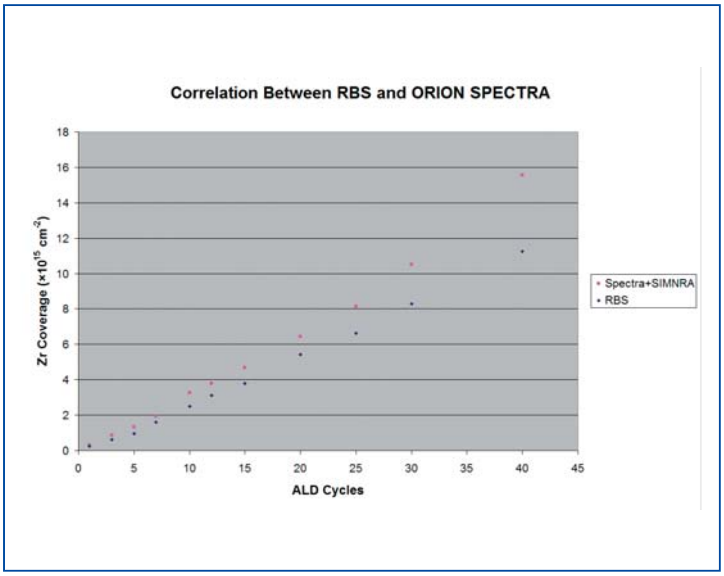

Figure 2: Correlation between RBS data and ORION® PLUS Spectra peak intensities. RBS analysis courtesy of Dr. Steffen Teichert, Qimonda (Dresden, Germany).

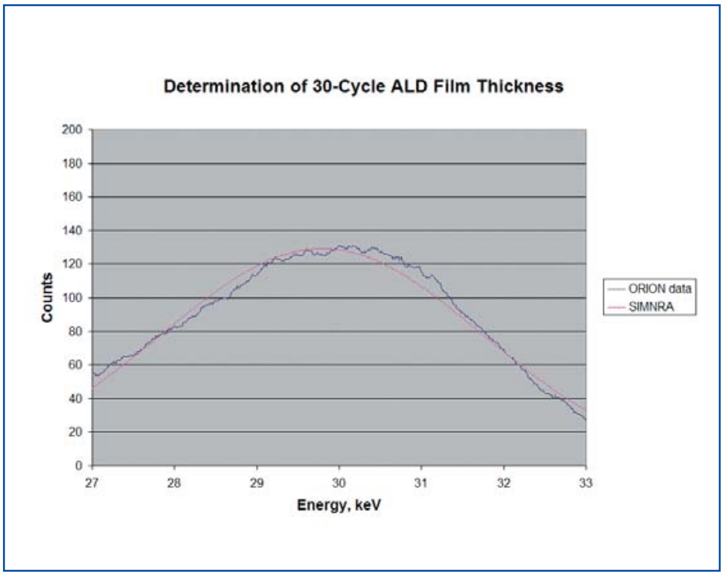

Figure 3: Peak fitting of the zirconium backscatter signal for the 30-cycle film.

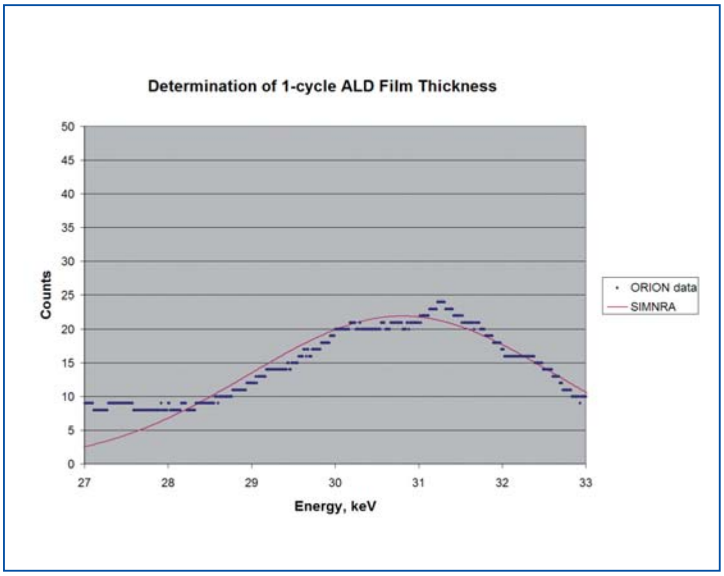

Figure 4: Peak fitting of the zirconium backscatter signal for the 1-cycle film.

To complete the analysis it is important to correlate the results to physical film coverage amounts. This is accomplished in the ORION® PLUS Spectra through the use of the simulation and data fitting software SIMNRA (developed by Matej Mayer at the Max Planck Institute for Plasma Physics). The program, originally intended for RBS and NRA measurements, can be extended to lower energy, with the assumption that its models remain valid. The sample information, geometries, and detector response function are input into the model. The program can then import experimental spectra and perform a fit. We focus the fit only on the channels of data corresponding to the zirconium peak. In this case this meant an energy range from 27-33 keV. The fit provided by SIMNRA (Figure 3) for the 30-cycle sample indicates that the film coverage is 3.5 × 1016 atoms/cm^2. Assuming the film to be a stoichiometric ZrO_2, this translates into a thickness of 12.1 nm.

To demonstrate the low detection limit in this application, we show also the analysis of the 1-cycle film. The analysis proceeds almost identically to that for the thicker film. The one adjustment necessary arise because, for this thinnest film, the presence of silicon in the model overwhelms the zirconium peak, making fitting impossible. Therefore we ignore the silicon edge in doing the fit in this case, and in fact do not include it in the model. As a check, we did model the 30-cycle film with and without silicon in the model, and we determined that the difference in film thickness predicted by the fitting only changed by 3 % when ignoring the silicon. Therefore we believe this simplification to be safely justified. Figure 4 shows the results of the fitting. Note that the count level is lower, as is to be expected. Also, the peak is shifted to higher energy; this occurs because there are less inelastic energy losses in a thinner film. There is simply less material for the ion beam to traverse !

In this case SIMNRA indicates the coverage to be 9.22 × 1014 atoms/cm^2. This translates into a film thickness of just 0.31 nm. This of course is a very reasonable result for a monolayer thick film.

In conclusion, it can be seen that the ORION® PLUS SPECTRA technology can be applied to thin film analysis in combination with microscopy, delivering a complete solution for demanding applications.

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Carl Zeiss NTS GmbH
Carl-Zeiss-Str. 56
73447 Oberkochen
Germany
Tel. +49 73 64 / 20 44 88
Fax +49 73 64 / 20 43 43
info@nts.zeiss.com

Carl Zeiss NTS, LLC
One Corporation Way
Peabody, MA 01960
USA
Tel. +1 978 / 826 1500
Fax +1 978 / 532 5696
info-usa@nts.zeiss.com

Carl Zeiss NTS Pte. Ltd.
50 Kaki Bukit Place #04-01
Singapore 415926
Singapore
Tel. +65 65 67 / 30 11
Fax +65 65 67 / 51 31
info.sea@nts.zeiss.com

Carl Zeiss NTS Ltd.
511 Coldhams Lane
Cambridge CB1 3JS
UK
Tel. +44 12 23 41 41 66
Fax +44 12 23 41 27 76
info-uk@nts.zeiss.com

Carl Zeiss NTS S.a.s.
Zone d'Activité des Peupliers
27, rue des Peupliers -
Bâtiment A
92000 Nanterre
France
Tel. +33 1 41 39 92 10
Fax +33 1 41 39 92 29
info-fr@nts.zeiss.com

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