Application Note

Elemental Nano-Volume Characterization of ALD Defect Particles by Auger Electron Spectroscopy

Overview

Advanced semiconductor manufacturing, following the trajectory of Moore's law, currently faces the unprecedented challenge of developing technology in the sub-10 nm node. One possible technology that may help in this task is Area Selective Deposition (ASD). ASD of features with sub-10 nm resolution can be achieved by controlling thin film deposition in "growth" regions, while inhibiting material deposition in "non-growth" regions.[2] Recent studies have demonstrated that such atomic scale manufacturing can be achieved via atomic layer deposition (ALD) followed by atomic later etching (ALE), a process that, when repeated in many cycles, allows for highly controllable area selective deposition.[3] The stability and reliability of this process can be profoundly degraded by unwanted nucleation in "non-growth" regions. These defects are typically too small for chemical analysis by low energy SEM-EDS. Knowing the defect composition is important information for designing a more selective ALD/ALE process. Here, Auger Electron Spectroscopy (AES) is used to chemically characterize such defect sites and therefore better understand the mechanism of selectivity loss during integrated ALD/ALE.

Sample Measurements

Low-temperature isothermal ALD/ALE cycles were used to deposit TiO_2 on SiO_2/Si patterned surfaces. Samples were created with N=12-14 cycles to produce ~8-10 nm thick TiO_2 in growth regions with some particles forming in the adjacent "non-growth" regions. These particles were analyzed using a PHI *710* Scanning Auger Nanoprobe equipped with a 25kV Schottky field emission electron gun and a coaxial Cylindrical Mirror Analyzer (CMA). The Auger instrument is equipped with an in-situ UHV compatible Oxford EDS detector. To minimize the effects of backscatter contribution in the data, EDS maps were acquired using 5 kV beam energy, while AES data was acquired at 25 kV.

Results

SEM analysis of the non-growth region reveals the presence of < 25 nm nanoparticles, indicative of selectivity loss (Figure 1 left). EDS Ti and Si maps of the defect particle region show no definitive particles present, while the particles are clearly visible in the AES Ti and Si maps (Figure 1 right). Titanium was below the detection limit of EDS in the non-growth area.

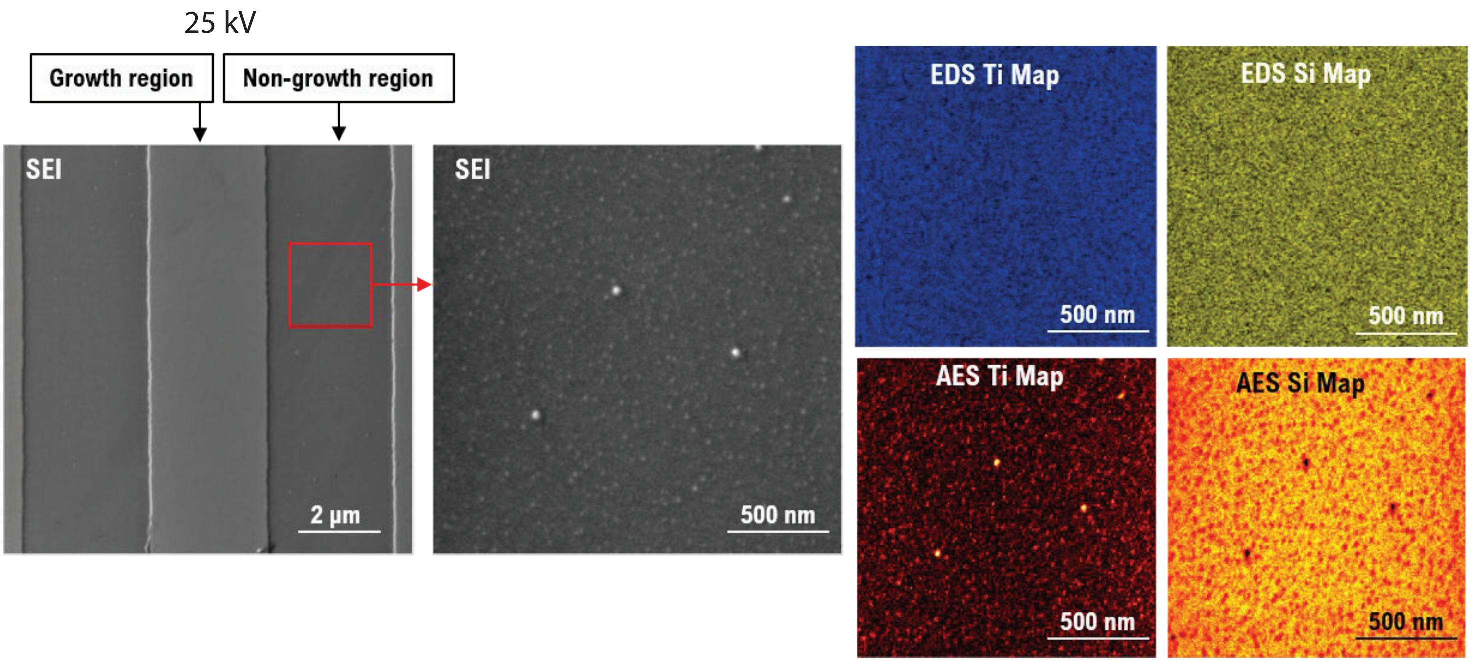
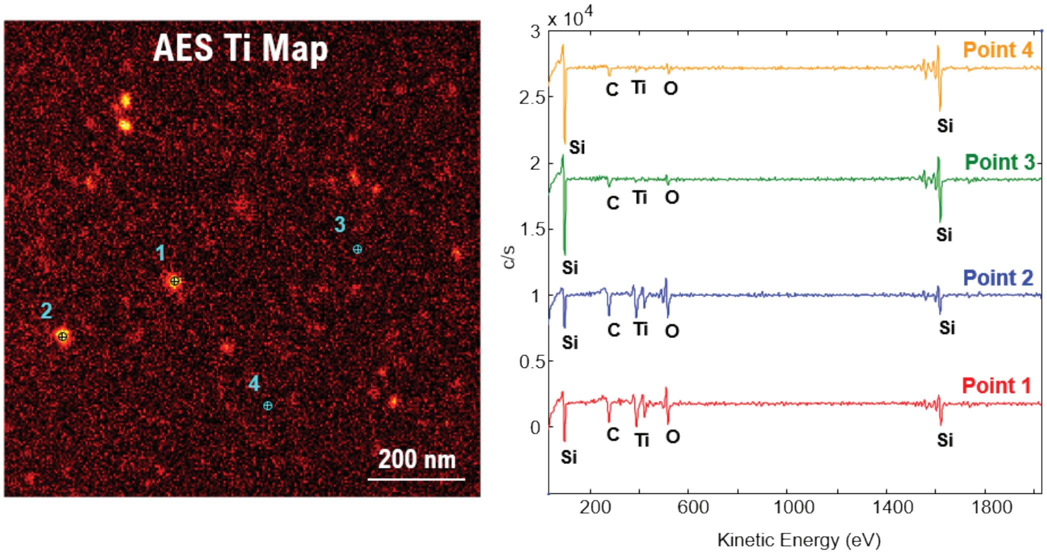


Figure 1. Secondary electron images of ASD sample and corresponding EDS and AES Ti and Si maps of non-growth region.

Using AES, the composition of the defect particles is readily quantified. In Figure 2, defect particles observed in the non-growth region were observed in the AES Ti map. Based on this map, four points were selected for quantification: two points on particles, and two points off particles.



	On particle		Off particle	
At. %	1	2	3	4
Si	38.7	34.5	81.8	80.0
Ti	15.6	15.9	2.5	3.1
O	15.7	17.1	4.3	3.9
C	30.0	32.5	11.5	13.0

Figure 2. AES Ti map (left) of non-growth region with four points selected for analysis. Auger point spectra (middle) and a table of the atomic percentages of detected elements (right) are shown.

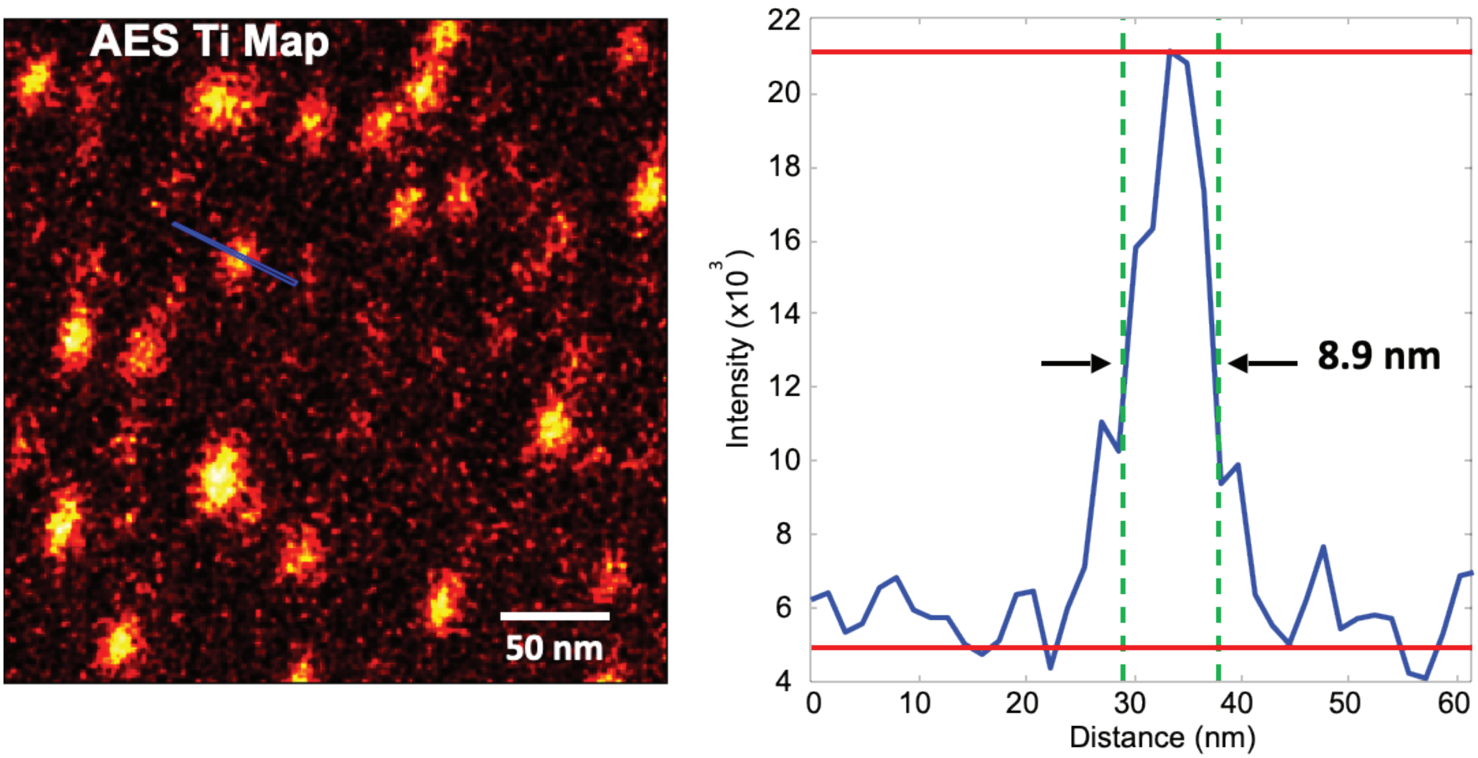


Figure 3. AES Ti map (left) with a line scan indicated in blue. The extracted line scan (right) is shown with an 8.9 nm particle diameter measured from the FWHM of the peak intensity.

In addition to chemical characterization and quantification of ALD/ALE defect particles, AES is also capable of accurately measuring nanoparticle diameters down to < 10 nm. In Figure 3, an AES Ti map was acquired from a 300 nm field of view. Several particles are present within the field of view. A line scan across a Ti particle (blue line in map) was extracted from the map and is shown on the right. From the extracted line scan, it is easily determined that the particle is 8.9 nm in diameter.

Conclusions

Selectivity loss is a major failure mechanism in the ALD/ALE area selective growth process. Understanding the chemistry and morphology of particles nucleating in non-growth regions is a critical step in mitigating such defects. Here, we demonstrate that Auger Electron Spectroscopy is a powerful technique for characterizing nanoparticles that form on the non-growth region of a TiO_2 film on a SiO_2/Si patterened substrate. These particles were found to consist of a higher Ti concentration (~15 atomic percent) as well as higher C (~30%) and O concentrations (~16%) compared to the underlying substrate. Transmission Electron Microscopy/Energy Dispersive X-ray Spectroscopy (TEM/EDS) analysis may also be able to characterize defect particles (in cross-section); however, the number of particles in a given Focused Ion Beam (FIB) cut will be limited. The top-down analysis of AES provides for a much simpler analysis with an almost endless number of particles that can be characterized. Furthermore, AES can be used to image particles < 10 nm in diameter to determine morphology, distribution, and size.

Acknowledgements

Physical Electronics would like to thank Seung Keun Song and Gregory N. Parsons at North Carolina State University, Department of Chemical and Biomolecular Engineering, for providing the samples.

References

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18725 Lake Drive East
Chanhassen, MN 55317, USA

www.phi.com
www.ulvac-phi.com

2500 Hagisono, Chigasaski
253-8522, Japan