



HIGH SPATIAL RESOLUTION AND HIGH ENERGY RESOLUTION AUGER DEPTH PROFILING of Ni/Si Films

OVERVIEW

The formation processes of nickel silicide thin films are of particular interest for sub-65 nanometer semiconductor manufacturing technologies. The formation of either Ni₂Si, NiSi or NiSi₂ depends primarily on the annealing temperature. The characterization of one dimensional and three dimensional structures formed under different annealing conditions is also of particular interest. Given the desire to characterize these silicide structures with nanometer scale spatial and depth resolution, scanning Auger spectroscopy combined with low energy ion beam depth profiling provides unique capabilities to compare the composition of films prepared under different annealing conditions. This application note demonstrates the ability of the PHI *700Xi* Scanning Auger NanoProbe to characterize a thin film of nickel deposited on a silicon wafer and annealed at 450 °C. The PHI *700Xi* Scanning Auger NanoProbe consists of a scanning electron gun with a minimum beam diameter of 7 nm and a coaxial Cylindrical Mirror Analyzer (CMA) for Auger spectroscopy, Auger imaging, and Auger depth profiling. A newly developed high energy resolution capability can be used to observe the chemical states in the Si and Ni Auger spectra while maintaining the unique Auger imaging capabilities of the CMA for imaging samples with topography.

EXPERIMENTAL PROCEDURE

A thin film of nickel was deposited on a silicon wafer and annealed at 450 $^{\circ}\mathrm{C}.$ A section of the sample



Figure 1: Sputter depth profile of an annealed nickel film on Si shown in the peak intensity display mode.

was mounted on the high energy resolution sample mount of the PHI 700Xi Scanning Auger NanoProbe. Secondary Electron Images (SEI's) were acquired with a 7 nm diameter electron beam (20 kV - 1 nA). Auger images were acquired with a 12 nm diameter electron beam (20 kV - 10 nA). Auger spectra were acquired with a 22 nm diameter electron beam (10 kV - 10 nA). Auger depth profiling was accomplished by alternately sputtering the sample with a 500 V - 0.6 uA argon ion beam rastered over a 1 by 0.5 mm area and collecting Auger spectra with a 10 kV - 10 nA electron beam. Chemical state depth profiles were calculated using the Linear Least Squares fitting software in PHI *MultiPak*. Spectral data was collected in either the standard mode (0.5%) or high energy resolution mode (0.1%) as indicated by the figure captions.

RESULTS

Figure 1 shows the N(E) peak intensity Auger depth profiles for Si and Ni acquired over a 20 by 20 micron area with 0.1% energy resolution.

The low intensity of Ni at the surface is attributed to carbon surface contamination which was quickly



Figure 2a: 0.1% High energy resolution spectra of Si KLL peaks in the Ni thin film and the Si wafer substrate.

Figure 2b: 0.1% high energy resolution spectra of Ni LMM peaks in the Ni thin film and the Si wafer substrate.

removed at the beginning of the depth profile. The interpretation of this relatively large area depth profile is that Ni and Si have interdiffused as a result of the annealing of the thin Ni film. Ni and Si spectra were extracted from the depth profile data set in the middle of the Ni film and near the end of the depth profile and are shown in Figures 2a and 2b. The 0.1% high energy resolution spectra show peak positions of the Si KLL peak for the silicon wafer at 1616.5 eV and for the silicide at 1617.2 eV. The peak positions of the Ni LMM were found to be at 846.2 eV in the Ni metal and at 844.8 eV in the Ni silicide. The data therefore suggests that the silicon diffused into the Ni film and is forming a relatively small amount of silicide and that Ni diffused into the Si wafer to form a similar small amount of Ni silicide.

Shown in Figure 3 is a SE image of the crater bottom after acquiring the sputter depth profile shown in Figure 1. Sub-micron islands are observed on the bottom of the crater. Figure 4 shows Auger spectra acquired from on (point 1) and off (point 2) an island. From the survey spectra and high

energy resolution spectra (not shown), the islands are Ni silicide and the crater bottom is clean silicon. These results indicate that the data of Figure 1 cannot be interpreted as the result of a simple onedimensional interdiffusion of Ni and Si. It is clear that three dimensional structures are being formed and it appears that the Ni silicide islands sputter at a slower rate than the surrounding Ni film on the silicon wafer.

A second sample was analyzed with high spatial resolution imaging and depth profiling. Ar⁺ sputtering was used to remove the approximately 12 nm of surface contamination detected in Figure 1. The SE image shown in Figure 5 reveals a heterogeneous surface structure. A color overlay display of the Auger images for C, Si and Ni acquired from the same area is shown in Figure 6. The Auger images clearly identify large areas composed of Ni and small features (200 to 500 nm areas) composed of Ni with Si.



Figure 3: SE image with a 5 μm field of view (FOV) showing the presence of sub-micron islands after obtaining the sputter depth profile shown in Figure 1.







Figure 5: SE image with a 5 μm FOV obtained from a new area on the sample after removing surface contamination with Ar sputtering.



Figure 6: Color overlay display of multiple Auger images: C (red), Ni (blue), and Si (green) showing the presence of 200-500 nm structures within the Ni film.



Multi-point depth profiles obtained with a 10 kV - 10 nA electron beam were acquired using 0.1% high energy resolution at point 1 (on the Ni film) and point 2 (on the Ni / Si structure). LLS fitting software was

Figure 7: Depth profile through the Ni film at point 1 in Figures 5 and 6. Nickel silicide is observed at the interface of the Ni film and the Si substrate



Figure 8: 0.1% high energy resolution spectra of the Si KLL and Ni LMM peaks showing the chemical shifts associated with nickel silicide formation at the interface of the depth profile in Figure 7.



Figure 9: The depth profile through a Ni/Si island at point 2 in Figures 5 and 6 reveals a complex silicide structure.

used to separate the Ni as metal and silicide as well as the Si as metal and silicide. The depth profile for point 1 is shown in Figure 7. Shown in Figure 8 are Si and Ni spectra from the depth profile obtained at point 1. The spectra show that the Si KLL peak shifts up in energy and the Ni LMM peak shifts down in energy at the interface region where a nickel silicide layer has formed.

The depth profile for point 2 is shown in Figure 9. As suggested by the overlay Auger images in Figure 6, the small structures are a mixture of Ni and Si. The use of 0.1% high energy resolution data provides the ability to separate the spectra of Si as silicide and silicon metal. The shape of the Si metal depth profile in figure 8 suggests that within the heterogeneous structure, there are regions that sputter at the same rate as the surrounding Ni film with a resulting apparent interface of 100 nm to the Si wafer and other silicide material that appears to sputter 3 times slower than the surrounding Ni film. This is supported by the appearance of islands in the SE image of Figure 3. These observed islands probably appear as a result of differential sputtering.

CONCLUSION

The formation of an interfacial NiSi (based on Auger quantification) is identified by PHI 700Xi Scanning Auger NanoProbe Auger depth profiling with high energy resolution. The use of Auger imaging with color overlay elemental displays and multipoint depth profiling at high spatial resolution identified additional Ni silicide nanostructures in the Ni film. Additional experiments using standard samples of different Ni silicide compositions can provide further accuracy for Auger quantification as well as measurements of the different sputter rates for metallic Ni, metallic Si, and multiple Ni silicide phases.

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