Bevel Depth Profiling SIMS for Analysis of Layer Structures

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Abstract. We are evaluating the use of bevel depth profiling Secondary Ion Mass Spectrometry (SIMS) for the characterization of layered semiconductor materials. In this procedure, a sub-degree angle bevel is cut into the analytical sample with an oxygen or cesium primary ion beam in a commercial SIMS instrument. The elemental distribution of the resulting bevel surface is then imaged with a focused ion beam in the same instrument. This approach offers maximum characterization of layered semiconductor materials. In this procedure, a sub-degree angle bevel is cut into the analytical sample with an oxygen or cesium primary ion beam in a commercial SIMS instrument. The elemental distribution of the resulting bevel surface is then imaged with a focused ion beam in the same instrument. This approach offers maximum characterization of layered semiconductor materials. In some cases, depth resolution can be greater than available from conventional depth profiling. Removal of residual surface damage/topography created during beveling has also been investigated by the cleaning of the bevel surfaces using gas-cluster ion beam sputtering before imaging analysis.

INTRODUCTION

Shrinking semiconductor device dimensions require the development of analytical approaches for increased depth resolution and the capability for 2-dimensional profiling. Bevel sectioning methods have been widely employed for compositional depth profiling. In this approach, a bevel is created through the thin film or multilayer structure to be examined. The beveled surface, with a bevel angle of $10^{-3}$ to $10^{-4}$ radians, provides lateral magnification of buried structures that are smaller than the depth resolution of the probing technique into lateral surface features that are larger than the lateral resolution of the technique. Subsequent high resolution imaging of the beveled surface reveals the elemental depth distribution of the material. Bevels can be produced in a variety of ways including mechanical polishing, chemical etching or ion beam sputtering. Bevel production by ion beam sputtering has become popular because it offers precise control of the beveling parameters (1). The use of ion sputtering for this application is also being driven by a growing interest in the use of Ga$^+$ focused ion beam systems for sample preparation (2). Ion sputtered bevel depth profiling has been employed for compositional depth profiling by Auger Electron Spectroscopy (Auger) (3), X-ray Photoelectron Spectroscopy (XPS) (4) and Secondary Ion Mass Spectrometry (SIMS) (2,5-7). There are several motivations for continued development of bevel depth profiling SIMS. Unlike conventional SIMS depth profiling, where sputtering and secondary ion analysis occur simultaneously, bevel depth profiling effectively decouples the sputtering and analysis steps. This decoupling provides the flexibility to optimize conditions for bevel production even when such conditions would degrade a conventional depth profile. The high lateral magnification offered by the bevel surface may allow small buried features to be resolved that are smaller than the depth resolution capabilities of current SIMS instruments. Another feature of beveling is that a single bevel sample can be examined by multiple complementary techniques with exact registration of analysis regions. These might include scanning electron microscopy (morphology), Auger (major element quantitative analysis) and SIMS (trace analysis). Finally, the use of bevel depth profiling provides both lateral and in-depth characterization and allows for "on-chip" analysis. In this work we examine the utility of using a commercial SIMS instrument to cut bevel structures into layered semiconductor materials with subsequent analysis of the bevels using microbeam imaging in the same instrument. We have investigated experimental parameters necessary to produce high quality bevel structures. We have evaluated the generation of 2-dimensional profile distributions from complex layered materials. Issues of surface roughness and ion beam mixing introduced during the beveling process are addressed through the use of post-processing of the bevel samples with a unique gaseous cluster ion source.

EXPERIMENTAL

Following earlier work by Hues et al. (6) and Merkulov et al. (7) bevel craters were created in a Cameca IMS 4F ion microscope*. An external, square-root raster waveform, produced by an HP arbitrary waveform generator (Model 33120A), was used as an external input for the X-direction raster signal. This waveform is shown in Figure 1. The external signal input (maximum 10 V peak-to-peak) is injected into the raster circuit of the ion microscope.

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before the input amplifiers. This allows the standard instrument raster amplitude settings to be used to control the size of the bevel raster. A Wavetech signal generator was used to input a free-running triangular-shaped waveform into the Y raster input. Overdriving either of the Cameca amplifier circuits with an input waveform at too high a frequency can result in distortion in the output waveform (See Figure 2). This distortion results in the beam dwelling for a longer time at the crater edges giving a distorted crater bottom. To avoid these effects, input frequencies of ~300 Hz were used in the X raster and 1-2 KHz for the Y raster. The size of the bevel crater was adjusted using a combination of the external input voltage and preset raster size controls on the instrument. Primary ion beam currents in the range of 10 nA - 100 nA were used for bevel cutting. In our previous work (6), imaging of the bevel was accomplished using a magnetic sector SIMS instrument in the ion microscope mode of analysis. This operational mode requires the use of higher primary ion current densities during imaging to produce adequate secondary ion signals. This had the deleterious effect of causing a shift of the layer position (and a loss in depth resolution) during image acquisition as the sample was sputtered. To minimize this effect, all images in the present study were acquired using static SIMS microbeam imaging with either a 10.0 keV impact energy \(\text{O}_2^+\) primary beam for positive secondary ion detection or a 14.5 keV impact energy \(\text{Cs}^+\) primary ion beam for negative secondary ion detection. Under static SIMS conditions, the primary ion beam dose absorbed by the sample during imaging is maintained below \(10^{13}\) ions/cm\(^2\) resulting in negligible sputter erosion of the sample and no shifting of the layers. Image acquisition was controlled using the instrument control software (Charles Evans and Associates, Redwood City, CA).

All image processing was conducted using Image Pro Plus (Media Cybernetics, Silver Spring, MD).

RESULTS

Bevel Crater Characterization

Bevels were first cut into silicon wafers to evaluate the linearity of the bevel and the lateral magnification under various conditions. Figure 3 shows a surface profilometer trace along the X (long) axis of a bevel cut into silicon. The slope of the bevel is controlled by modification of the input voltage to the X raster input and by the sputtered depth. Input voltages of 2-9 V peak-to-peak were used for the bevel rasters. Bevel craters are rectangular in shape with a bevel (X-axis) length of ~200 \(\mu\)m - 500 \(\mu\)m and Y axis length of 100 \(\mu\)m - 250 \(\mu\)m. Higher voltages produced shallower bevel craters. Bevel angles were in the range of \(~9\times10^{-3}\) radians - \(2\times10^{-4}\) radians which gave lateral magnifications which we could vary from a factor of \(~100-5000\). At the higher magnifications, a depth feature of 1 \(\text{nm}\) would be magnified to about \(~5\) \(\mu\)m in the bevel projection. This is larger than the lateral spatial resolution of the SIMS instrument which we estimate to be \(~0.5\) \(\mu\)m. Greater magnifications may be possible, but conditions were selected to keep the bevel structure within the imaging field-of-view of the instrument which is limited to 500 \(\mu\)m x 500 \(\mu\)m. To test the ability of the bevel approach to resolve thin buried layers, a Ge delta-doped test structure in silicon was analyzed. The sample consists of 5 Ge delta spikes buried 0.2 \(\mu\)m below the silicon surface. Figure 4 shows a secondary ion image and line profile of a bevel cut with a 10.5 keV impact \(\text{O}_2^+\) primary ion beam. The spacing between delta layers is ~40 nm and each layer is clearly resolved.

FIGURE 3. Surface profilometer trace of a bevel crater in silicon along the X (bevel) axis.
One significant advantage of bevel profiling SIMS is the flexibility to decouple the beveling conditions from the actual sample analysis. Optimal conditions for beveling may not be favorable for analysis and vice versa. For example, Figure 5 shows two bevels cut into a depth profiling reference material. This sample is composed of 7 Ta₂O₅ layers separated by delta SiO₂ layers (8). The thickness of the Ta₂O₅ layers ranges from 18.0 nm to 19.0 nm and the SiO₂ layers are about 1.0 nm in thickness. To monitor the location of the delta layer, we could use Cs⁺ sputtering with detection of SiO₂ negative secondary ions (the sample is biased negatively for this mode of operation). This would be a standard condition used for depth profiling of negative secondary ions in a magnetic sector SIMS instrument. Cs⁺ bombardment enhances the yields for negative secondary ions providing maximum detection sensitivity. Unfortunately, the use of the positive Cs⁺ ion beam with a negatively biased sample results in a high impact energy for the Cs⁺ (14.5 keV) resulting in degraded depth resolution. However, since the beveling step is decoupled from secondary ion analysis, bevels can be produced using Cs⁺ and a positive sample bias giving an impact energy of 5.5 keV. These conditions reduce atomic mixing, even though such a setup would not be favorable for analysis in a conventional profile. Subsequent imaging analysis with a high energy Cs⁺ microbeam is then used to analyze the bevel structure. Figure 5 shows a comparison of the bevel produced using “standard conditions” (5a) compared to the lower energy bevel (5b). Significant improvement in depth resolution is indicated by the increased peak-to-valley ratios of the individual layers.

Another use of bevel depth profiling SIMS is for analysis of samples where both lateral and in-depth information must be acquired. The lateral wet-thermal oxidation of high aluminum content AlGaAs layers on silicon can be used to form buried oxide layers which are widely employed for the fabrication of vertical cavity surface emitting laser structures (VCSEL’s). These devices are being developed for many applications including communications systems and optical interconnects. We have employed bevel depth profiling SIMS to characterize MBE grown, laterally oxidized, Al₀ᵦGₐ₀₂As layers in an attempt to determine compositional uniformity through the oxide layers, and the lateral extent of oxidation. Typical samples for this study consist of alternating layers of AlₓGa₁₋ₓAs and GaAs. Each AlGaAs layer is ~80.0 nm thick and is separated from adjacent layers by ~150 nm of GaAs. The surface is capped with ~200 nm of GaAs. The aluminum composition, x, was varied from 0.90 to 1.0 to determine the effect on the lateral oxidation rate. Each sample has a series of trenches etched through the film to expose the buried AlGaAs layers. Trenches are spaced ~100 µm apart. Oxidation is achieved by wet oxidation in a specially constructed furnace. The lateral extent of oxidation was designed to be ~25 µm from the trench. A microbeam SIMS image looking down at the trench and the lateral oxidation front is shown in Figure 6a.

Initial attempts to conduct depth profiles in the laterally oxidized areas gave highly distorted depth profiles in the vicinity of the trench edge. As shown in Figure 6(b), bevel profiling using Cs⁺ bombardment revealed simultaneously the in-depth variation and lateral penetration of the different oxide layers. However, the variable lateral penetration of the oxidation front and severe sputter rate differences between the oxidized layers and the GaAs layers tended to make interpretation of the images problematic.

To minimize distorting effects, bevels were cut into the oxidized AlGaAs samples using Cs⁺, O⁺ and Ga⁺ primary ion beams at several different energies. Both Cs⁺ and Ga⁺ beveling produced very distorted bevels resulting from the large sputter rate differences between oxidized and unoxidized layers and a high

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**Figure 5a.** Secondary ion image and associated line scan of SiO₂⁻ distributions from a bevel crater cut through a series of 7 SiO₂ delta layers in Ta₂O₅ using 14.5 keV Cs⁺. **Figure 5b.** Same sample with bevel cut using 5.5 keV Cs⁺. Vertical scale on the line scans is counts/pixel.

**Figure 6a.** Secondary ion image of O⁺ from laterally oxidized AlGaAs structure. **Figure 6b.** 14.5 keV Cs⁺ bevel crater cut into oxidized sample showing lateral and in-depth distribution of oxidation.
degree of surface topography as determined by SEM imaging. Best results were obtained by using O\textsuperscript{2+} bombardment for bevel production. Using higher energy O\textsuperscript{2+} beveling (19.5 keV impact), in an effort to produce higher levels of implanted primary ion beam oxygen (to increase analytical sensitivity for subsequent imaging), produced significant sample topography. Oxygen backfilling did not appear to offer any significant advantage in this situation. 3 keV O\textsuperscript{2+} impact sputtering produced the highest quality bevel craters with minimal sample topography and layer shift. A summary of the results from this comparison is shown by the series of SEM images in Figure 7.

Other Examples of Bevel Depth Profiling

We have applied the bevel depth profiling technique to examine a variety of other materials including multilayer metal films, delta-doped layers in silicon, ultra thin oxides, nitrides and zirconium films on silicon and patterned wafers. Direct analysis of bevel cross sections “on-chip” is potentially a very attractive approach for depth profiling of small, complex and non-planar structures. Figure 8a shows a O\textsuperscript{+} SIMS image of a bond pad on silicon coated with 0.4 µm of copper. A cross sectional bevel has been cut in the bond pad using 10.5 keV O\textsuperscript{2+}. This sample is used by SEMATCH to test various copper damascene processes. The dark region in the lower part of the image shows the location of the bevel crater. Figure 8b is a higher magnification Si\textsuperscript{+} SIMS image showing the distribution of the silicon under the bond pad. This work is continuing to develop optimal bevel conditions to minimize surface topography during bevel formation and to evaluate copper diffusion into the silicon and the efficacy of Ta diffusion barriers in these materials.

Evaluation of Gaseous Cluster Beam Sputtering for Surface Cleaning of Bevels

As with conventional depth profiling, bevel profiling is limited by the development of sample topography and ion beam mixing effects, both of which are magnified by the bevel structure. Also, in some samples, the use of an optimal bevel cutting beam for reduction of topography may compromise the ability to do subsequent imaging analysis. For example, oxygen beveling was found to minimize distortions in the AlGaAs samples, but the beveled surface is implanted with oxygen from the primary ion beam which masks the true oxygen distribution in the sample. Skinner was the first to address these issues for Auger bevel analysis by using a second, low-energy sputtering step to remove beam-induced mixing produced by the higher energy beveling beam (3). McPhail utilized chemical etching of a beveled surface to remove the oxide layer produced by the bevel cutting beam (5). In the present study we have conducted preliminary experiments to address the issue of residual mixing/roughness by post bevel processing of the samples with a gas cluster ion beam (GCIB) (9). The GCIB is a beam containing 1000-5000 argon, oxygen or nitrogen molecules in an ionized cluster held together by weak Van Der Waals forces. The impact energy of each constituent of the cluster is ~ 10 eV, leading (potentially) to virtually no penetration of the cluster below the surface. Bombardment of silicon surfaces using these clusters has been demonstrated to provide a high degree of surface smoothing. We hope that post bevel processing by GCIB cleaning will remove residual surface topography and ion beam mixing effects while introducing no additional degradation of the sample.

For this experiment we beveled a depth profiling reference material with repeating monolayers of CrO\textsubscript{2} spaced 30 nm apart in Cr. The sample was beveled with
a 14.5 keV Cs+ ion beam. The O SIMS image of the beveled but unprocessed sample is shown in Figure 9(a) along with its associated line scan. Degradation of the depth resolution of the layers is apparent as the bevel gets deeper from left to right in the image. In this example, the degradation results primarily from sample topography as observed by SEM. Figure 9b shows a similar sample, in this case bombarded after beveling with the gaseous cluster beam using a dose of \(10^{14}\) cluster ions/cm\(^2\). A noticeable improvement in our ability to resolve the layers is noted. It should be noted that the GCIB cleaning tends to leave a thin oxide layer on the surface. This was removed in this example by a short Cs sputter clean. Atomic Force Microscopy of the bevel crater bottom in the area of the deepest layer demonstrated an RMS roughness that decreased from 25 nm in the unprocessed sample to 6-8 nm for the cluster bombarded sample. Further improvement would be expected with more aggressive sputtering in the GCIB system. Ongoing studies will attempt to determine appropriate cluster doses for optimal smoothing and ion beam damage removal.

**CONCLUSION**

Bevel depth profiling offers several advantages for depth profiling analysis. Decoupling of ion beam sputtering and secondary ion analysis provides flexibility to modify the sputtering conditions to achieve the best possible depth resolution. Bevel profiling offers the possibility of 2-dimensional profiling of dopants and buried interfaces. The wide variety of beam species, energies and incident angles available on a commercial SIMS instrument makes it a very flexible tool for bevel production. Compared to conventional depth profiling, a limitation of bevel depth profiling is that the secondary ion signal for the species of interest is spread out over many thousands of pixels in an image. This leads to poor statistics for an individual pixel and an overall reduction in sensitivity. Partial solutions to this issue involve using advanced image processing algorithms to optimize the signal while maintaining an acceptable spatial resolution. We are also participating in the development and testing of a high brightness, small spot size, oxygen ion source that may be useful for increased spatial resolution imaging with higher sensitivity. The use of GCIB processing of bevel structures to remove implanted primary ion beam species, ion beam mixing effects and surface topography is especially attractive. To fully exploit this technology, further research is needed to develop experimental conditions to maximize the potential advantages of this approach.

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**REFERENCES**


*Certain commercial equipment, instruments or materials are identified in this paper to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.