Characterization of Nanostructures Micromachined with Focused Ion Beams (FIB)

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Abstract

Focused Ion Beam (FIB) technology has been demonstrated to be a powerful technique in micro-machining and micro-fabrication. As the critical dimensions of the device migrate towards the nanometre regime, FIB becomes an important tool in the construction of these devices. In machining and fabrication with FIB in the deep submicron dimensions, an important consideration is the ability to correlate the structures with the processes of deposition or milling so that the instrumental parameters can be optimized. Effects such redeposition during milling in such fine dimensions will have to be controlled. However, since feature size of these structures fabricated with the FIB often have profiles of a few 10's of nanometres, it is increasingly difficult to use standard analytical techniques such as scanning electron microscopy (SEM) to resolve such features. At the same time, atomic force microscopy (AFM) with it's near atomic resolution is now a standard metrology tool in semiconductor manufacturing.

In this paper the authors would like to demonstrate, using Atomic Force Microscopy (AFM), the effects of parameters such as probe current on the micromachined profiles produced by FIB. We will compare these results obtained with AFM with those using SEM. Control of these parameters in the fabrication of nanostructures on different substrate materials such as metals and semiconductors will also be demonstrated.

Keywords

Focused Ion Beam (FIB), Atomic Force Microscopy, Micromachining, Microfabrication

Introduction

Focused ion beam (FIB) micromachining and focused ion beam deposition/microfabrication enable maskless patterning and processing of materials at extremely high resolution. This technology has the potential applications in high volume microstructure fabrication as well as in the more limited applications such as prototype construction and low-volume, high-value specialty devices. Since the late 1970's FIB technology has found a wide range of applications in the microelectronics industry, from three-dimensional device testing and proof-of-concept, failure analysis cross sectioning, to lithographic mask repair and integrated circuit modifications. In the late 1980's several research thrusts were pursued to establish FIB as a useful technique in microstructure fabrication. Many novel applications emerge such as using the FIB as direct-write tool for micromachining¹, machining of micron size gears^{2,3} and microsurgical tools⁴. Although most of the current

micromachined features are in the micron size, focused ion beam has also been successfully implemented to produce 100nm small apertures for near-field scanning optical microscopy^{5,6}.

As the critical dimensions of the device migrate towards the nanometre regime, prototyping in a standard processing environment becomes increasingly expensive. Although FIB has the limitation of creating only one structure at a time, nevertheless, the technique can be used to produce small quantities of customized structures which otherwise would require substantial investments to produce, e.g. microelectromechanical (MEMS) lithographic/etch technologies. Once the viability of the prototype is established, alternative methods of mass production may then be considered. With a combination of direct-write and etching techniques at extremely high resolution, FIB could prove to be a very useful tool in prototyping nanostructures. To achieve this, it is essential to understand the interaction of the ion beam with the processed substrate materials. It is also important to characterize FIB processes to identify in advance possible pit falls and necessary enhancements. Material characteristics, such as surface roughness, as characterized by the modern techniques such as AFM, should be well understood..

Experimental Details

50nm of Tungsten or Titanium thin films were sputtered onto Silicon [111] wafers with a Denton model SJ/24LL RF sputter coater. Similarly 50nm Nickel or Titanium thin films were evaporated onto Silicon [111] wafers using a Denton model SJ/20C e-beam evaporator. Bare silicon wafers were used as experimental control. A FEI 200xP FIB system with a Ga⁺ liquid ion source was used for the direct-write and etch of nanometre lines. The resultant features were characterized with a Digital Instruments Multimode Scanning Probe Microscope Nanoscope IIIa, a JEOL 6300F and a Philips XL30 scanning electron microscope. Figure 1 illustrates the pattern used for our FIB processing characterization studies. The ion beam parameters listed were programmed into the FIB workstation. Different probe currents and different milling depths were used for each of the four quadrants, with the lines closest to the pattern axis having the sharpest focus. After focusing the ion beam before the commencement of each experiment, the probe was slightly defocused while etching across the pattern.

Results and Discussion

An initial experiment was performed on a Silicon [111] control wafer to ascertain the effect of the FIB ion beam current on the profiles of the etched trenches. Figure 2 is an AFM line scan profile across three submicron lines etched into the silicon substrate. The FIB currents for the lines are 11, 4 and 1 pA and the depth control was 1 μ m. Since the focused ion beam current was preset for a certain milling depth, with increasing beam current, the dwell time of the beam is expected to decrease accordingly. Thus the depths of these nano-trenches should decrease with increasing current. Interestingly, from the scan, the 1pA etched line exhibits the most material re-deposition at the edge of the trench. As the current increases, material build up becomes less prominent. Typically material being sputtered away from a surface has a cosine dependence⁸ for the spatial distribution of ions leaving the surface. As the beam is scanned across the surface, the angle of incidence is decreased. The ion beam forms a moving front with a slope angled with respect to the beam. This is the etch front in the material. This leads to material being deposited on the surface instead of being removed by the pumping system because the cosine distribution is now tilted towards the back in the reverse direction of the beam scanning direction. (Figure 3) The reduction in the material build up with increasing current could be due to the fact that there is a greater rate of sputtering at higher current. Large scale rectangular features milled in silicon⁷ have used serpentine scanning procedures to help eliminate

the material re-deposition associated with deep trench milling. However, with the nano trenches using 1pA and 4pA, the high aspect ratios of the trenches may require other methodologies in order to completely remove material redeposition.

The two low ion beam probe currents of 1 and 4 pA were chosen after the initial etching experiment to further investigate the effects of the ion beam probe currents on different substrates. Figure 4a and 4b are SEM images of 1pA and 4pA FIB nano trenches in nickel thin film. The tightly focused beams produce clean etched profiles, as expected. As shown, the nickel film has been completely etched to the silicon substrate. The bright bands along the sides of the trenches are caused by the sloping of the trench edges. The effects of defocusing of the ion beam on the profiles of the nano trenches were also investigated. Figure 5a and 5b show the corresponding trenches using defocused 1pA and 4pA probe currents. The defocused trenches are broad with incomplete material removal. The higher probe current has removed more material when defocused, however material removal is still incomplete.

Figure 6a and 6b compare the line profiles obtained when the AFM is operated in contact mode vs. in tapping mode. Clearly higher resolution in both the trench depth and trench profile can be achieved with the tapping mode. This is due to the smaller probe radius (~ 1-5 nm) used in tapping mode, compared with the larger probe radius used in contact mode (~20-50 nm). Operating the AFM in tapping mode provides greater lateral resolution and reduces smearing effects which may occur when using contact mode. However the disadvantage in using AFM in tapping mode is the slow scanning speed and thus the longer experimental time.

Figures 7 and 8 are the corresponding AFM tapping mode line scans for the SEM 1 pA and 4 pA nano trenches shown in Figures 4 and 5. The focused trenches have clean sharp profiles, whereas the defocused profiles show material still present at the trench bottom.

Figure 9 is an AFM line scan across four nano trenches on a silicon substrate etched with a 1 pA probe current and depth control of 0.05 μm . As the probe is defocused from right to left, the trench profiles become smeared. At the farthest right hand trench where the beam is the most defocused, instead of etching trench, deposition of material actually was observed. This could be due to the two side walls building up and joining together. This trend can also be seen in the second etched line where the deposition has broaden from the tightly focused first line. In addition to re-deposition of etched materials, surface contamination could also be a cause of the build up of material at the trenches. In imaging using a SEM with small stationary probe, contamination spot is often observed. The spot is made up of hydrocarbons from the vacuum system and the substrate surface. Because of the low ion current used, the sputter rate is not sufficient to remove the material from the substrate and contaminates migrate to the probe location, resulting in the material build up as observed.

Further investigation of FIB processing reveals a profile dependence of the nano-trenches on the surface roughness of these metallic thin films. The surface roughness was measured with AFM and is summarized in Table 1. Line scans of the trenches etched in silicon (Fig.6), nickel (Fig.7), tungsten (Fig.10) and titanium (Fig.11) with a 1pA focused ion beam were compared. Silicon substrate has the smoothest surface with a RMS value of 0.121nm and the titanium film has the roughest surface with a RMS value of 2.06 nm. As the roughness of the surface increases, the minimum feature which could be resolved decreases. Re-deposition on the edges of the trenches decreases as the surface roughness increases. With the roughening of the surface, there is a continually varying angle of incident to the ion beam. Thus the distribution of the sputtered ions varies accordingly. The sputter ions which do contribute to the re-deposition now see a highly contoured surface to be filled in. Under the low pA ion currents used in these experiments, ion beam sputtering time was not sufficient to completely fill up the rough surface and therefore sharp profiles similar to those observed in the silicon sample could also be found in the titanium samples. As the beam was defocused, the etched trenches become less well defined. Figure 12a is a SEM overview of the silicon etched pattern and Figure 12b is that of the tungsten etched pattern. Interestingly the etched lines using 1pA ion current are clearly visible in tungsten (upper left hand quadrant) but almost invisible in silicon. This is not due to the FIB definitions of the trenches on these surfaces. Instead when viewing with SEM, the enhanced surface roughness in tungsten, with the increase in surface topographical changes, enhances the visibility of the etched lines under SEM.

Conclusion

Nano trenches were fabricated with Focused Ion Beam (FIB) system. Both AFM and SEM were used to characterize the profiles of these etched lines. In AFM tapping mode produces better depth profiles than contact mode, but with a reduced scanning speed. It was also found that in these small dimensions, viewing the nano trenches with SEM only sometimes could be misleading. It is necessary to define the trench profiles with atomic resolution AFM measurements. From our experimental observations, nano trenches etched with small ion beam currents are dependent on the substrate materials. Redeposition of the sputtered material on the edges of the trenches is also substrate dependent. Polycrystalline thin films influence the nature of the profile due to the enhanced surface roughness, whereas for single crystal surfaces, significant redeposition of the etched materials is observed. A tightly focused ion beam produces sharp, well defined profiles in both silicon and metallic thin films, and with the defocusing of the ion beams, broadening of the etched profiles was observed. The use of gas assisted etching is now being explored to improve the etched profiles of these nano trenches.

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Figure 1. Ion beam parameters used to produce the pattern etched into the thin films.

Figure 2. Trenches etched into silicon. FIB currents are 11, 4, 1 pA. Depth control 1 μm. AFM in contact mode.

Figure 3. Diagram indicating the development of a an advancing slope front in the material being milled ⁸.

a) b)
Figure 4. a) 1 pA trench in nickel with a focused ion beam, b) 4 pA trench in nickel with a focused ion beam. Both trenches have a depth control of 0.5 μm .

a) b)
Figure 5. a) 1 pA trench in nickel with a defocused ion beam, b) 4 pA trench in nickel with a defocused ion beam. Both trenches have a depth control of 0.5 μm .

a) b)
Figure 6. a) Focused 1 pA nano trench in silicon, AFM in contact mode, b) Focused 1 pA nano trench in silicon. AFM in tapping mode. Depth control was 0.05 μm .

Figure 7 a) Focused 1 pA nano trench in nickel, 0.5 μm , tapping mode, b) Focused 4 pA nano trench in nickel, 0.5 μm , tapping mode.

Figure 8 a) Defocused 1 pA nano trench in nickel, 0.5 μm , tapping mode, b) Defocused 4 pA nano trench in nickel, 0.5 μm , tapping mode.

Figure 9. Defocused series of 1 pA nano trenches in silicon. AFM in contact mode. Depth control was 0.05 μm .

Figure 10. a) Focused 4 pA trench in tungsten, b) focused 1 pA trench in tungsten

Figure 11. a) AFM line scan for titanium for focused 1pA probe current b) Corresponding 3D image of the surface showing the surface roughness. Z scale is 40 nm per division.

a)

b)

Figure 12. a) Over view of the silicon etched pattern, b) Overview of the tungsten etched pattern.

Material	Roughness RMS (nm)	Line Widths (nm) for 1 pA lines
Silicon	0.121	162
Nickel	0.230	128
Tungsten	0.503	53
Titanium	2.060	63

Table 1. Summary of surface roughness values measured and the corresponding trench widths using AFM.