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#### The effects of dwell time on focused ion beam machining of silicon

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#### Keywords

Focused ion beams, Ion implantation damage, Dwell time, Raman spectroscopy

#### Abstract

In this study, the effects of dwell time on Ga<sup>+</sup> focused ion beam machining at 30 keV for different milling currents were investigated. The surface topographies were analysed using atomic force microscopy (AFM) and the substrate structures were investigated by means of Raman spectroscopy. It has been observed that by increasing dwell time the total sputtering yield was increased even though the total dose was remained the same. Also the silicon damage by ion bombardment is reduced as the dwell time is increased. This is mainly due to catalyst behaviour of Ga inside Si which over a period of hours causes recrystallization of Si at room temperature by lowering the activation energy for crystallization.

#### 1. Introduction

Focused ion beam (FIB) systems are widely used as a versatile tool for nanofabrication prototyping, device modification and ion beam lithography. Two main categories of FIB micromachining are sputtering and deposition. FIB sputtering is a maskless microfabrication technique where incident ions transfer momentum to the substrate and release atoms through cascades of collisions.

Since material modifications and patterning have to be controlled from micro to nano-scale, a thorough understanding of the fundamental mechanisms involved in FIB machining is required to enable full use of the opportunities offered by FIB [1]. The total dose which is usually expressed in terms of ions/cm<sup>2</sup> is the most significant parameter for FIB milling and dose-related effects in sputtering of semiconductors such as Si have been reported [1]. In particular, FIB generated surface topographies for the doses relevant to the early stages of FIB milling have been studied [2]. Damage effects in Si in particular was investigated by Spoldi et al [3].

However, other parameters in addition to dose, notably pixel dwell time, affect final geometry, while unintentional damage due to ion bombardment occurs to the substrate. During line by line scanning, the ion beam pauses on a particular point (dwell point or pixel point) for a predetermined time (dwell time) and then moves to the next pixel point. There have been relatively few studies of the effect of dwell time and those investigated high doses, several orders of magnitude above the threshold dose for milling. For example, it has been reported that for doses of the order 10<sup>18</sup> ions/cm<sup>2</sup> there is a significant increase in sputtering yield at elevated substrate temperature [1].

In this paper we investigate the effect of dwell time on Si sputtering and associated sub surface damage for a small dose of 10<sup>18</sup> ions/cm<sup>2</sup> of Ga ions used for FIB milling at beam currents of 100pA, 300pA, 500pA and 1000pA. The milling depth in each case was analysed by atomic force microscopy (AFM) and for investigation of Si structural damage Raman spectroscopy was employed.

#### 2. Theory

FIB can be used for direct and maskless patterning of substrates. It induces secondary processes such as recoil and sputtering of constituent atoms, defect formation, electron excitation and emission, and photon emission [4]. Sputtering is defined as the removal of atoms from a solid surface due to energetic particle bombardment [5]. Associated with this process, ion implantation changes the target composition to the depth below the surface at which the incoming Ga ion stops. Therefore the density of the target and the nuclear and electronic stopping of projectiles and recoils are changed locally where the incoming ions land.

For sputtering with normal incidence, Sigmund's theory enables calculation of the sputtering yield assuming negligible re-deposition [6]. It is important to note that the depth distribution of the implanted Ga ions is independent of the ion flux [7]. In the Sigmund model, the target temperature does not have any direct effect on sputtering yield [8]. However, increased substrate temperatures may speed up the dynamic annealing process and make the substrate more crystalline.

Sigmund's Theory can be summarized by the following equations:

$$Y(E) = \frac{0.042}{U_s} \alpha(\frac{m_i}{m_t}) S_n(E)$$
(1)

$$S_n = 4\pi a Z_i e Z_t e \frac{m_i}{m_i + m_t} S_n(\varepsilon)$$
 (2)

$$a = 0.468 (Z_{i}^{\frac{5}{3}} + Z_{t}^{\frac{5}{3}})^{-\frac{1}{2}}$$
(3)  
$$\varepsilon = \frac{m_{t}}{m_{i} + m_{t}} \frac{a}{Z_{i} e Z_{t} e} E$$
(4)

Where, Y(E) is sputtering yield (atoms/ion) at normal incident, E ions energy (keV),  $U_0$  the atomic binding energy (eV),  $m_i$  atomic mass of incident ion (amu),  $m_t$  atomic mass of target atom (amu), a is the efficiency of energy transfer, and  $Z_i$ ,  $Z_t$  are nuclear charge of incident ion and target atom respectively.

The ion implantation simulations were carried out using SRIM software [9] (Figure 1).



Figure 1. Ion implantation trajectories of Ga in Si at 30 keV

The mean stopping range of ion implantation for 30keV Ga<sup>+</sup> ions is 27nm and the implantation profile can be considered as a Gaussian distribution in depth z.

#### 3. Experimental

A dual-beam FIB machine, the Strata DB 235 (FEI, UK) was used to perform micromilling of (100) n-doped Si. The ion beam is generated from a Ga liquid metal ion source and is accelerated to 30keV. The field of view (FoV) is divided into 4096×4096 pixels. The actual step size for the beam depends on selected overlap, magnification and beam current. A total of 12 squares each measuring  $5\mu$ m ×  $5\mu$ m were milled at four different currents, *viz.* 100pA, 300pA, 500pA, 1000pA and three different pixel dwell times of 0.1 µs, 1µs and 10µs were tested. Figure 1 shows the arrangement of milled squares. An overlap of 50% and magnification of 10,000X were chosen for milling the squares. The step size between pixels for 100pA and 300pA was 7.4nm and for 500pA and 1000pA it was 14.8nm. The chamber pressure during the milling was  $10^{-6}$  mbar. As each beam current selection aperture gives different focus and beam offset, after switching between currents, an area outside the milling regions was chosen to focus the beam in order to avoiding unintentional damage in the experimental region. The applied dose was  $10^{17}$  ions/cm<sup>2</sup> for each square.

The topographies of the implanted areas were acquired using a NanoWizard II atomic force microscope (JPK Instruments, UK) operating in intermittent contact mode at a tip velocity of 2  $\mu$ m/s, employing pyramidal tipped Si cantilevers (PPP-NCL, Windsor Scientific, UK). Figure 2 shows a typical 3D topography measurement from these AFM measurements. For investigation of the Si crystallinity, Raman measurements using a Renishaw Raman Microscope with 514 nm laser edge, 1800 I/mm grating and the laser power of 0.36 mW were carried out. The measurements were obtained by averaging three scans with a resolution of 1.5 cm<sup>-1</sup>.



Figure 2. 3D AFM measurements for surface topography

#### 4. Results and discussion

Figure 3 shows the milling depth at different currents for chosen dwell time. It was observed for all currents that by increasing the dwell time the sputtering yield is increased. Sputtering of a target by energetic ions or recoil atoms is assumed to result from cascades of atomic collisions. Sufficiently high energies and fluence cause at first a crystal-to-amorphous transformation and ultimately material removal. The differences in depth of milled regions receiving the same total dose but for different pixel dwell times is explained as follows. As dwell time increases, the ion beam spends more time at each pixel site causing more ion implantation and damage occurs on the substrate beneath. From Monte Carlo simulations (Figure 1) it is observed that the volume of implanted regions is about 1000nm<sup>3</sup>. As dwell time increases, more ions are accumulated in this volume (de-channelling) and more damage occurs to the substrate produce more amorphous Si. As the amorphous Si has weaker bonds, considering eq.1, the sputtering yield is increased. Also the density of the damaged volume increases increasing the likelihood of sputtering per pixel "pulse" by dint of the higher number of loosely bound atoms present in the amorphized and densified pixel region. As dwell time decreases, fewer ions are implanted and less damage is created. Therefore when the beam moves to next pixel, dynamic annealing occurs to greater extent [10].



Figure 3. Effect of dwell time on milling depth at milling currents in the range 100-1,000pA

Raman results show that by increasing the pixel dwell time the ratio between crystalline Si and amorphous Si is increased.

By increasing the dwell time, more ions are implanted. Therefore more damage is created and the substrate becomes more amorphous for each beam pulse. More amorphous substrate and more heavy implanted ions would increase the de-changeling effect [11]. Also by increasing the number of implanted Ga ions within the same volume, the chance of initiating the precipitates is increased [12]. For shorter dwell times a less amorphous substrate is created and dynamic annealing is applied to greater extent as the substrate is less damaged, when compared to longer dwell times. This would cause greater mobility of defects and diffusion of implanted Ga, leading to short range recombination such as Frenkel pair annihilation, as well as rearrangement of defect complexes within the region of a collision cascade [7].

It has been observed that Ga species can act as a catalyst for crystallization of Si at room temperature by decreasing the value of activation barrier  $\Delta G_{act}$  [13]. Therefore, increasing the dwell time increases the probability of nucleation of Ga inside the substrate. This would create densified Ga regions which promote greater recrystallization of Si at room temperature over a period of several hours after the implantation is complete. Consequently for similar total doses, the regions milled with longer dwell times have more crystalline Si due to having a higher density of Ga nucleation. Figure 4 shows Raman spectra for 100pA; similar behaviour was observed for other currents.



Figure 4. Raman measurements at 100 pA for three different pixel dwell times at a fixed dose of  $10^{17}$  ions/cm<sup>2</sup>

#### 5. Conclusions

The effect of changing pixel dwell time in Ga FIB milling of a crystalline Si surface was studied experimentally using AFM to measure topographical changes and Raman spectroscopy to investigate effects on crystallinity, all for a fixed dose of ions. It was observed that higher dwell times cause less damage to the Si substrate while increasing the sputter yield. This behaviour is explained by detailed consideration of the ion implantation process in the presence of dynamic annealing. In order to achieve better understanding of substrate structure at different dwell times further studies including transmission electron microscopy measurements are desirable.

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