Progress towards site-specific dopant profiling in the scanning electron microscope

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Abstract. 2-dimensional dopant mapping in the scanning electron microscope is based on the variation of contrast as a function of differences in the number of active dopant atoms. It was put forward as a potential technique for dopant mapping on the nano-scale in response to the challenge of ever-shrinking device dimensions. Its impact, so far, has been limited as the majority of the work has concentrated on sample preparation by cleaving, thus not achieving the site-specificity which is required for nano-scale electronic devices. It was previously shown that cross-sections prepared using a 30 kV gallium focused ion beam (FIB), results in such strongly reduced dopant contrast (DC) that quantification is impossible. We demonstrate that by the use of a final in-situ argon broad ion beam (BIB) milling operation, contrast from a FIB prepared cross-section can be greatly improved. As a result, SEM dopant mapping could find a much more wide-spread application.

1. Introduction
As electronic devices and components shrink in size, the challenge of characterization of their dopant distributions becomes ever more intense. Scanning electron microscopy (SEM) has the potential to solve this challenge by the use of so-called dopant contrast (DC). DC has been known for many years [1] but received increased interest after the publication of results obtained with a field emission gun SEM which is required for successful imaging using DC [2].

The ultimate aim of a dopant mapping technique is to be able to map the location of the dopants within a device in 3 dimensions. Previous work into SEM DC has focused on the characterization of cleaved surfaces. This method of sample preparation has a major drawback; it does not allow site specific investigation of nano-scale devices. One technique which has the ability to create site-specific cross-sections is the focused ion beam (FIB). It was previously shown that FIB preparation of a silicon surface reduced the attainable contrast to levels insufficient for reliable analysis and incomparable to that obtained from a cleaved surface [3]. It was suggested that the reason for this was that the escape depth of secondary electrons (SEs) was only marginally greater than the depth of the amorphous damage caused by the FIB milling. Other dopant mapping techniques which rely on FIB preparation have also reported such effects. For example, users of electron holography have reported an electrically dead zone in a silicon p-n junction to a depth of ~25 nm [4] as well as an amorphous layer.
Many studies into the effect of final FIB energy on damage produced have been conducted [5, 6, 7] and it has been shown that a reduction of FIB energy can reduce damage depth to as little as 1.5 nm [8] after the use of a 2 kV gallium ion beam. To date, a reduction of damage layer thickness has not yet been reported to improve SEM DC.

This paper presents results of the use of a low energy (1 kV) argon ion miller in-situ inside the FIB as a final polishing step to improve the dopant contrast from conventionally prepared FIB cross-sections of doped silicon test structures. We show that the argon ion miller can improve contrast provided that the size of the trench is taken into consideration.

2. Experimental

Two silicon test structures have been produced: Sample 1 is a p-n junction structure produced by chemical vapour deposition (CVD) and Sample 2 is a doping level test structure produced using molecular beam epitaxy (MBE). Sample 1 consists of a 2.5 µm thick boron doped layer (5×10$^{18}$ atoms cm$^{-3}$) upon an arsenic doped (1×10$^{19}$ atoms cm$^{-3}$) substrate wafer. Sample 2 consists of 7 boron doped silicon layers with a constant thickness (200 nm) with doping levels reducing from 5×10$^{19}$ cm$^{-3}$ to 4×10$^{15}$ cm$^{-3}$ upon an arsenic doped (1×10$^{19}$ cm$^{-3}$) substrate. Each layer was separated by a spacer layer and the highest doped layer was situated directly adjacent to the substrate wafer.

Cross-sections were prepared using an FEI Helios with a 30 kV gallium ion beam using successively lower beam currents. The beam energy was then reduced to 10, 5 and 2 kV for polishing; images were collected after each milling step. Imaging was carried out using immersion mode, a 1 kV primary electron beam energy, through-lens detector, a mirror bias of -15 V, a tube bias of 70 V and a working distance of 5 mm.

For the in-situ argon milling experiments, standard FIB cross-sections were polished using an Oxford Applied Research LiOn 50 in-situ argon broad ion beam (BIB) miller attached to a Carl Zeiss XB1540 FIB. The BIB energy was 1 kV with an incidence angle of 18.5° to the FIB milled surface. The resulting cross-sections were imaged using a primary electron beam energy of 2 kV, an in-lens detector and a working distance of 5 mm.

To compare the DC to the extent of ion beam damage, expected amorphous layer thicknesses on silicon were calculated by generating stopping / range tables using SRIM. The lateral straggle length was then used to calculate the expected amorphous layer thickness using the expression $T = 3 \Delta R_t$, where $T$ = amorphous layer thickness and $\Delta R_t$ = lateral straggle length as proposed by Wang et al [9].

3. Results and Discussion

Figure 1(a) is a plot of contrast obtained from within a trench cut perpendicular to the p-n junction using varying gallium ion accelerating voltages. The general trend is that as the ion beam energy decreases, the contrast increases. In comparison to this, a plot of the expected amorphous layer thickness is presented which shows the opposite trend. These results demonstrate that the amorphous layer thickness is of utmost importance when considering dopant contrast from FIB prepared surfaces. This is also true for studies using electron holography techniques. Here it has been noticed that, as well as the amorphous layer, an electrically “dead zone” is present on silicon p-n junctions to a depth of 25 nm which causes a dramatic reduction in the measured potential at the junction within this zone [4]. As SEM DC is produced by active dopants [10], dopants in this “dead zone” will make little contribution to the DC obtained. As the damage depth is expected to be reduced when using a lower energy ion beam, a greater proportion of detected SEs will originate from the undamaged silicon and contribute to the dopant contrast. It can be seen from Figure 1(b) that contrast does not substantially increase until a layer thickness of ~10 nm is achieved. This suggests that the majority of SEs which contribute to DC originate from a depth of < 10 nm.

In order to further reduce the amorphous layer and electrically damaged zone thickness produced by the preparation of the cross-section, a low energy, in-situ argon BIB was used. It has been shown by a number of authors that the application of BIB milling after FIB preparation of TEM thin foils can have a dramatic effect on the damage layer thickness [11, 12].
Barna et al. [11] showed that by the use of a BIB of 0.25 kV, the amorphous damage depth in silicon can be reduced to as little as 1 nm. Langford et al. [12] compared TEM lattice images of silicon before and after BIB milling and found the image sharpness and contrast to be greatly increased after BIB milling; this is indicative of a reduction in sample damage when compared to conventional FIB preparation. As well as the advantages of reduced damage layer thickness, the use of argon ions also reduces any possible effect of implanting gallium (a dopant atom for silicon) into the silicon target.

Figure 2 shows images of the p-n cross-section produced after 30 kV gallium milling and 20 minutes of in-situ BIB milling together with the contrast profiles from these cross-sections. The contrast present after gallium ion milling only is approximately 5.5 %. When the same cross-section is exposed to 20 minutes of argon BIB milling, the contrast (for the same imaging conditions) is increased to approximately 11.5 %.

This procedure has also been applied to multilayer Sample 2 (Figure 3). The observed contrast values are hugely reduced for FIB specimens when compared to a freshly cleaved specimen, and the contrast from the layers does not seem to be dependent on their doping level. Although the application of BIB milling does slightly improve the contrast obtained, the contrast of the lower doped layers still does not correspond to their doping level. The trench size may be affect the detection efficiency and hence lead to uneven dopant contrast from the prepared cross-section. In order to assess the effect of...
the FIB trench size, a larger trench was produced. The contrast profiles from the larger trench show a more expected trend; the highest doped layers have the greatest contrast which decreases with reducing doping level (Figure 3(b)). The large trench also produces an increased contrast. The application of argon milling improves the max. contrast level of the cross-section from ~4.5% to ~7%.

4. Conclusion
It has been demonstrated that together with careful consideration of the trench geometry, the application of in-situ argon broad ion beam milling can dramatically improve the dopant contrast attainable from a FIB prepared cross-section. These results represent a major step towards achieving site-specific dopant profiling of real nano-scale semiconductor devices.

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