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2014 J. Phys.: Conf. Ser. 522 012049

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Helium ion microscopy and energy selective scanning electron microscopy – two advanced microscopy techniques with complementary applications

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Abstract. Both scanning electron microscopes (SEM) and helium ion microscopes (HeIM) are based on the same principle of a charged particle beam scanning across the surface and generating secondary electrons (SEs) to form images. However, there is a pronounced difference in the energy spectra of the emitted secondary electrons emitted as result of electron or helium ion impact. We have previously presented evidence that this also translates to differences in the information depth through the analysis of dopant contrast in doped silicon structures in both SEM and HeIM. Here, it is now shown how secondary electron emission spectra (SES) and their relation to depth of origin of SE can be experimentally exploited through the use of energy filtering (EF) in low voltage SEM (LV-SEM) to access bulk information from surfaces covered by damage or contamination layers. From the current understanding of the SES in HeIM it is not expected that EF will be as effective in HeIM but an alternative that can be used for some materials to access bulk information is presented.

1. Introduction

The field of scanning electron microscopy (SEM) has seen major changes in the past decade in particular in terms of resolution improvements for low accelerating voltages, e.g. monochromated LVSEM resulting in sub-nanometer resolution [1]. Also a novel microscope was introduced, the Helium Ion microscope, HeIM, (in which the electron gun is replaced by a helium ion source), with an ultimate expected focused spot size of 0.25nm [2]. With such progress in lateral resolution, new application areas for both SEM and HeIM are opened up and it is increasingly recognized that interpretation of contrast in SEM images needs more attention.

While there have been some successful attempts to quantify SEM contrast for specific applications microscopes, it is also recognised that the contrast obtained from different instruments varies widely [3]. Often this has been blamed on the variation of secondary electron yield with the surface condition, i.e. the presence of oxide or carbon contamination layers. Of course this has to be considered as a possible explanation, especially since it has been found in both HeIM [4] and LVSEM [5] that organic mono-layers lead to strong contrast, albeit the exact mechanism is a matter of debate with several attempts to link contrast based on SE yield to differences in functional groups, surface potential,



surface energy and molecular ordering. While very useful for the imaging of self-assembled organic mono-layers (SAMs), it raises the issue of uncertainty in SEM contrast interpretation as result of surface layers. Further doubt on any attempts of quantitative SEM image contrast arises from the observation that contrast is very strongly influenced by the particular detector used in one instrument or different instruments. Given this situation one could come to the conclusion that there is no hope in understanding SEM contrast because of the limited information collected in the SEM.

One solution is to move away from the sole focus of SE yields (intensities) and start to consider the additional information contained within the SE emission spectrum (SES). Therefore, here SES are considered in more detail and the ways that they can be exploited for a better informed interpretation of image contrast are explored. In addition, differences in SES found in HeIM and LVSEM and the consequences for accessing bulk information or obtaining the best possible resolution are considered.

2. The secondary electron spectrum caused by low energy ELECTRON impact – accessing bulk information

Unlike electron energy loss spectra (EELS), secondary electron spectra (SES) are not widely known to have very distinctive features, perhaps due to the fact that SEMs are often operated at accelerating voltages of 20kV. Nevertheless, coincidence spectroscopy between EELS and SES has revealed a clear link between them [6]. It is therefore not surprising that SES collected using LVSEM (<5kV), where collision cascades are short and localised, differ sufficiently enough for fingerprint identification of inorganic materials based on the LVSEM SES spectra [7]. A more topical example demonstrating the importance of SES is that of graphene covered Ni in comparison to bare Ni. While the yield of the graphene covered Ni and the bare Ni were found to be similar, very marked differences in the LV SES for these two cases were reported [8]. Further experiments of varying accelerating voltage (250eV-900eV) on the graphene covered Ni show very distinctive changes in the SES, whereby the low energy peak starts to dominate with increasing accelerating voltage, reminiscent of the electron cascade, that leads to SE generation in the bulk resulting in SE with low energy when reaching the surface [8]. Detailed Monte Carlo calculations of the SES of this system were not reported but Monte Carlo modelling of SiO₂ also demonstrates that the high energy end of the SES is dominated by SEs generated at the surface while a substantial fraction of low energy SEs are likely to have been excited closer to the bulk (deeper than 10nm in the case of SiO₂) rather than at the surface [9]. Figure 1 (field of view, FV 6 μm) demonstrates that this increased escape depth for low energy SEs can be used to recover bulk information on a damaged surface, using dopant contrast as an example.

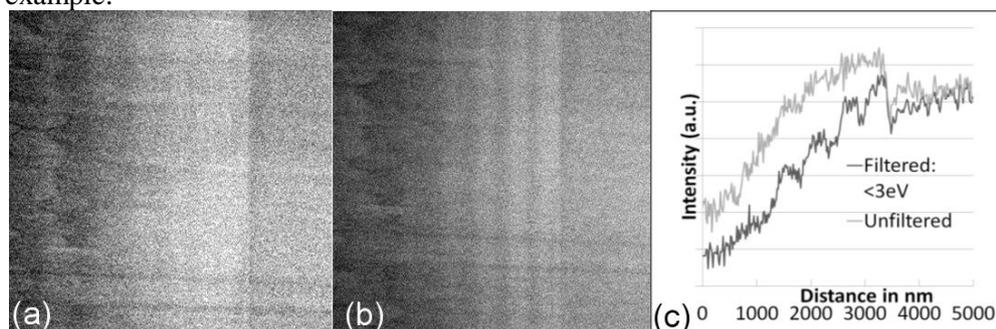


Figure 1 LV SEM (a) p-doped layers in Si not separated after Ga ion exposure, (b) as (a) but SE < 3eV only (c) Intensity profiles, from (a) and (b)

In LVSEM p-doped silicon layers show clear contrast on cleaved specimens but individual layers are not resolved when the sample surface is damaged during focused ion beam (FIB) preparation using 10 kV Gallium ions at grazing incidence. This can be understood as a result of the lateral displacement of dopant atoms, originally confined to the doped layers, by up to ~25nm, according to TRIM simulations. EF- imaging of such a Si surface does not improve the situation because the maximum escape depth, even for low energy SEs, is still located within the damage layer. However, if the same specimen is kept in air or exposed to an oxygen containing plasma, oxidation occurs from the surface,

which also contains the most heavily ion beam damaged layer (with majority of damage located within of $\sim 5\text{nm}$ from the surface, as determined by TRIM). The escape depth of the low energy SEs is increased in grown SiO_2 compared to Si and now exceeds 10nm , deeper than the majority of the Ga-ion damage. This makes no difference to dopant contrast of a standard (unfiltered) SE image which is strongly dominated by SEs originating from the damaged layer. Hence the individual doped layers still cannot be distinguished (Figure 1a). However, when only low energy SEs ($<3\text{eV}$) are detected, the individual layers can be resolved again, as the image is now predominantly formed from SEs originating from below the heavily ion beam damaged material. Figure (1c) contains intensity profiles that demonstrate the above more clearly.

This filtering approach also works for other materials systems as demonstrated in Figure 2 (a,b). The material is a triblock copolymer. In the standard SEM image in Figure (2a) the presence of details is obscured and contrast is dominated by topography as result of microtome damage. When only SE with energies $<8.3\text{eV}$ are used to form the image, fine detail appears (Figure 2b), best seen in the higher magnification inset. This demonstrates again the power of LVSEM in combination with EF in accessing bulk information. We note that the best suited cut-off energy depends on the material investigated based on the experimental or modelled SES.

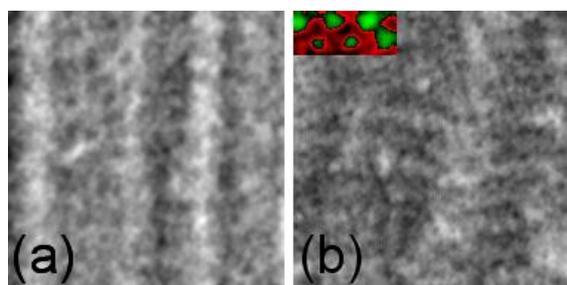


Figure 2 LV SEM images taken at 1keV primary beam, Tube bias 250V , Working distance 3mm : (a) stained tri-block polymer (FV 500nm), the vertical lines are sample preparation artefact (microtome damage), (b) as (a) but energy cut off for SE set at 8.3eV , the damage related features are much less pronounced, instead horizontal chains become visible. The inset shows chain at higher magnification (width 94nm).

3. The secondary electron spectrum caused by HELIUM ION impact – accessing surface information

The HeIM is renowned for its high chemical contrast and high lateral resolution. Figure 3a is a HeIM image from the same material, shown in Figures 2. While Figure 2b suffers from a limited lateral resolution, Figure 3a has limited chemical information, in spite of the expected better lateral resolution. It is assumed that this lack of chemical information is a result of the extreme surface sensitivity of HeIM. Evidence for this can be found by comparing the contrast at p-n junctions with varying concentrations of dopant atoms in the HeIM and LVSEM. This contrast is related to the potential difference V_{pns} between the p and n doped regions at the surface. V_{pns} is expected to reach a value determined by the doping concentration differences through the Fermi level alignment, V_{pnFA} if no surface states were present. In the presence of surface states (expected for silicon cleaved in air), V_{pns} only gradually reaches the value of V_{pnFA} , when moving from the surface into the specimen. The depth of this surface band bending region becomes shallower, with increased doping levels and can explain a sharp increase in doping contrast in the LVSEM for doping levels $>10^{18}\text{cm}^{-3}$ [10,11]. The sharp increase in contrast is a result of the surface band bending depth becoming smaller than the SE escape depth. This phenomenon can also be observed in the HeIM, but at a higher doping level of 10^{19}cm^{-3} [11], and hence a shallower band bending region. The latter suggest a shallower escape depth for SEs generated by He ion impact when compared to those generated by electron impact, leading to extreme surface sensitivity.

Further evidence for the enhanced surface sensitivity is found by the collection of SES generated by 30kV He ion impact [12]. These SES show a shift of the peak energy to smaller energies as well as a reduction in the width of the SES peak. Based on the description of the relationship between the depth of SE origin and SE energy in section 2, it can be concluded that the energy of the most energetic SEs generated at the surface by HeIM impact differs from that resulting from electron impact. If we

assume this also applies to the internally He-ion generated SEs then the escape depth is affected and for carbon it is only ~ 0.5 of that for electron impact according to the data in [13].

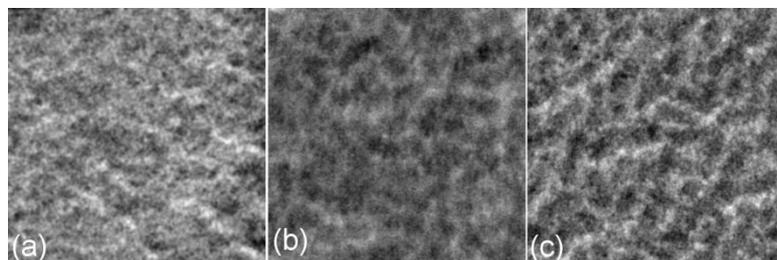


Figure 3 HeIM images): (a) stained tri coblock polymer field of view 300nm, (b) P3HT:PCBM polymer blend as received field of view 150nm, (c) same specimen as (b) after 3 min of plasma cleaning (process gas is air).

Based on the above discussion, HeIM will have less scope for making use of energy filtering for the purpose of accessing bulk information covered by damage layers due to the smaller maximum SE escape depth involved. Of course, we should recognise that it is precisely the latter that contributes to the excellent lateral resolution and surface sensitivity of HeIM SE imaging.

An alternative solution to access bulk information on some material in the HeIM may be the use of a plasma to etch away surface layers as demonstrated in [14] and in Figure 3(b,c). Figure 3b shows the as-received surface of a P3HT based bulk heterojunction solar cell material imaged in the HeIM at 30keV but the contrast becomes clearer when P3HT rich surface layer is removed through plasma etching.

4. Conclusion

Many development efforts in microscopy have been devoted to improving lateral resolution, but a practical limitation often encountered for advanced SEM is how to gain information representative of the bulk material in the presence of limited contrast due to damage or contamination layers. It has been demonstrated here that the filtering of the relatively broad SES in LVSEM can address this challenge effectively, while HeIM is best suited for applications where extreme surface sensitivity is paramount. Energy filtering in HeIM provides less scope for access to bulk information as a result of a reduced SE escaped depth but under some circumstances, a plasma can be used to remove surface layers.

Acknowledgments

The authors thank M. Tureau & T. H. Epps (College of Engineering University of Delaware) for providing the tri block polymer samples and A. J. Pearson and D. G. Lidzey Department of Physics and Astronomy, University of Sheffield for providing the P3HT:PCBM polymer blend sample.

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