

## The characterization of delta doping by fresnel contrast analysis

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**ABSTRACT:** Quantitative information about a dopant profile is notoriously difficult to obtain for nanometre thick layers using any technique currently available. Here Fresnel Contrast Analysis is applied to the characterisation of a boron delta doped layer in silicon. It is demonstrated that the approach can be applied accurately despite the presence of absorption. For a well localised layer less than 1at% boron in silicon can be profiled using this technique.

### 1. INTRODUCTION

Layers of dopant atoms in a semiconducting material can be incorporated during growth as two dimensional sheets, nominally only a single atomic layer thick. Such 'delta doped' layers remain important as quasi 2D electron gas systems, both for device applications and for fundamental studies. The accurate modelling of electronic behaviour in such systems relies on a knowledge of layer compositions to monolayer accuracy but this is currently impossible using any of the established techniques. Compositional data obtained using SIMS and X-ray diffraction are usually, for example, either too qualitative or too indirectly related to the actual layer. Sub-nm layers are usually only visible using standard high-resolution and CTEM techniques where clustering of dopant atoms has occurred or high concentrations of dopant have spread over many unit cells. However, it should be possible to obtain quantitative information about the dopant profile of layers containing concentrations of less than a tenth of an atomic layer ( $10^{13}$  atoms  $\text{cm}^{-2}$ ) using Fresnel Contrast Analysis in the TEM. The absence of visible Fresnel contrast at a given imaging condition is also useful in the provision of a lower limit for the diffuseness of such a doped layer. As a demonstration of how the approach can be applied we assess here the diffuseness of a nominally delta doped boron layer in silicon. The lower limit of the concentrations in such layers which can be detected using the technique is also investigated.

### 2. RESULTS OF FRESNEL METHOD APPRAISAL OF BORON $\delta$ -DOPING

The delta doped layer examined here was grown on {001} silicon with a nominal boron sheet density of  $1.6 \times 10^{14}$   $\text{cm}^{-2}$  (24 at%), as measured from the area under a SIMS profile. This is more than two orders of magnitude higher than the solubility limit at the growth temperature and corresponds to a change in the scattering potential relative to Si of  $\Delta V = 1.7\text{V}$ , assuming no change in the silicon lattice parameter. Boron atoms are expected to be fully substitutional for the low growth temperature of  $480^\circ\text{C}$  used. This rather high concentration should enable successful profiling even if the layer has diffused during growth over several unit cells. The experimental methods used and the general approach needed for the appraisal of Fresnel contrast have been described elsewhere (e.g. Ross and Stobbs 1991a). The Fresnel series examined comprised 13 plates and was taken using a JEOL 2000FX, a  $\text{LaB}_6$  filament and a fairly large objective aperture (Airy disc radius 0.18nm). The beam convergence was 0.3mrad and the crystal was tilted to a systematic row  $5.7^\circ$  from {110}. Accurate defoci were fitted to the images using power spectra from the contamination at the specimen edge, and the defocus step size between images was calculated to be 208nm. Particular care was taken experimentally both to ensure that the edge-on specimens examined had the layering vertical and to maintain a low contamination level (which causes background phase contrast noise). Accordingly, the specimen surface was cleaned

using 2kV Ar ions, and the Fresnel series was taken at an accelerating voltage of 100kV (below the threshold voltage of 145kV for knock-on damage in silicon). Full atomic multislice simulations were computed to simulate the experimental image series using consistent parameters with  $C_s=2.3\text{nm}$ , a focal spread of 30nm (the experimental energy spread is uncertain but makes little difference to the Fresnel contrast) and a sampling of 0.012nm/pixel.

Examples of images from the series are shown in Fig.1. The crystal thickness at the position of the boron layer was measured to be 100nm using weak-beam thickness fringes. Every second plate was digitised and the images were scaled to provide absolute electron intensities. The low signal to noise ratio necessitated careful projection of each image over a length of 50nm to minimise contributions from random noise. From the measured incident electron intensity, it is disturbing to note that 50% of electrons are lost in the image at the specimen edge despite the considerable care taken with specimen preparation and the minimisation of radiation damage in the microscope. The can only be attributed to ion damage and contamination caused during specimen preparation, but fortunately we can compensate for absorption caused by events outside the crystal by dividing the intensity profile near the interlayer by a linear background. Fig.2a shows the original profiles, divided by a linear background and projected over 50nm of the layering. Absorption due to the dopant is visible at zero defocus, indicating that contrast caused by electron diffraction out of the aperture and phase contrast effects are of comparable importance. Given the low atomic number of B relative to that of Si the greater absorption of B must be related to the diffuse scattering associated with local site irregularities and this is consistent with the way such layers exhibit bright contrast in High Angle Dark Field (HADF) images (e.g. Perovic et al 1991). Similar relative differences in absorption effects were seen for Fresnel contrast image series of GaAs / AlAs multilayers by Ross and Stobbs (1991b) who demonstrated that they could be dealt with successfully by dividing the entire series by the in-focus profile. A similar approach was used here, and the resultant profiles are shown in Fig. 2b. The more familiar form of Fresnel contrast is now evident. For a weakly diffracting orientation, as used here, we can ignore any asymmetry in the profiles when measuring an average profile and the symmetrised versions of 2a and 2b shown in Figs 2c and 2d were in the main used in the assessment of the doping profile. Small contrast features in these profiles are artefacts of the image processing, and we concentrate on comparing the more gross experimental and theoretical fringe contrast and spacing as defined in Fig.2d. Graphs of the experimental fringe contrast and spacing are shown in Fig.3 for the original profiles after they had been divided by the in-focus image. This division, taking account of differential absorption, leads to a shift of the minimum contrast back to zero defocus and it then remains to note the low experimental contrast values for which the approach can then be applied.

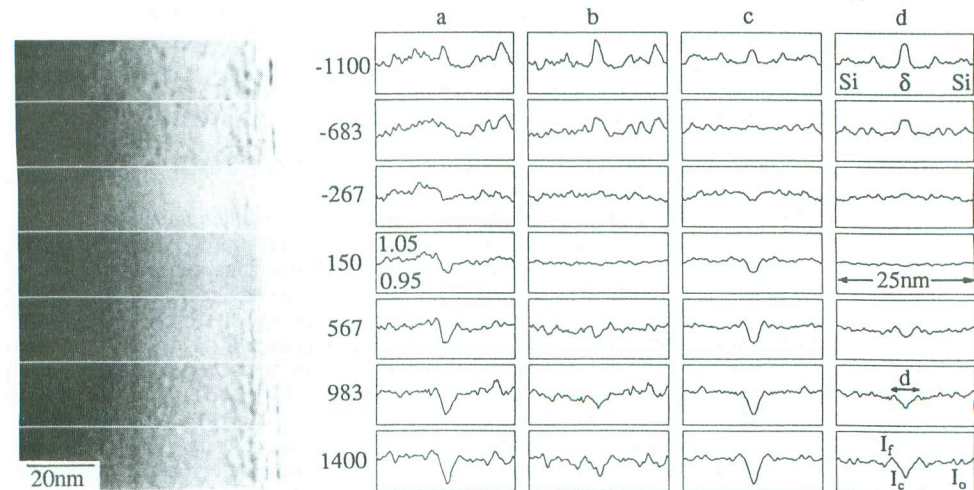


Fig.1 Images from a through-focal series of a boron delta doped layer in silicon at the defoci shown (nm).

Fig.2 Digitised Fresnel fringe profiles at the defoci shown (nm) a) averaged over 50nm parallel to the layer; b) now divided by the in-focus image; c) and d) are symmetrised versions of a) and b). The symbols  $I_c$ ,  $I_f$ ,  $I_o$  and  $d$  are defined in d).

It is useful to remember that when matching simulations with experimental data it is a profile for the mean forward scattering potential that is being fitted, and this can be affected both by the compositional profile and by a change in the lattice parameter of the interlayer. Let us look firstly at the effect of changing the dopant profile (assuming it to be completely substitutional), retaining throughout the lattice parameter of the 'matrix' silicon. Figs.4a, b and c respectively show profiles for 100, 24 and 2.4 at% B confined to a single atomic sheet. The contrast values at 2.4 at% are considerably greater than that exhibited by our experimental data. This demonstrates that, if there is no change in the silicon lattice parameter associated with the presence of the dopant, then a true delta layer containing less than 2 at% boron could easily be profiled. If we now assume the integrated sheet density of 24 at% boron as measured using SIMS to be correct (though further confirmation using high-angle contrast would seem advisable), the effect of spreading the boron *evenly* over 8 and 20 atomic layers can be seen in Figs. 4d and 4e. The corresponding graphs of fringe contrast and spacing are plotted together with the experimental graphs on the same scale in Fig. 5. It is clear from the width of the best-fitting potential profile that the boron must have diffused over at least 2nm. The simulated contrast, however, is obviously too high, especially close to zero defocus. Neither interstitial atoms nor carbon or oxygen contamination can be responsible, as these would both increase the contrast.

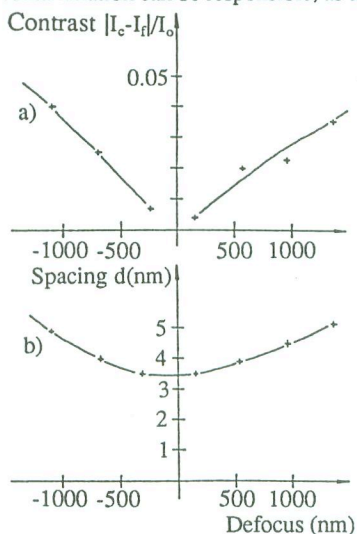


Fig.3 Experiment: a) first fringe contrast; b) first fringe spacing vs defocus for the profiles of Fig.2 d).

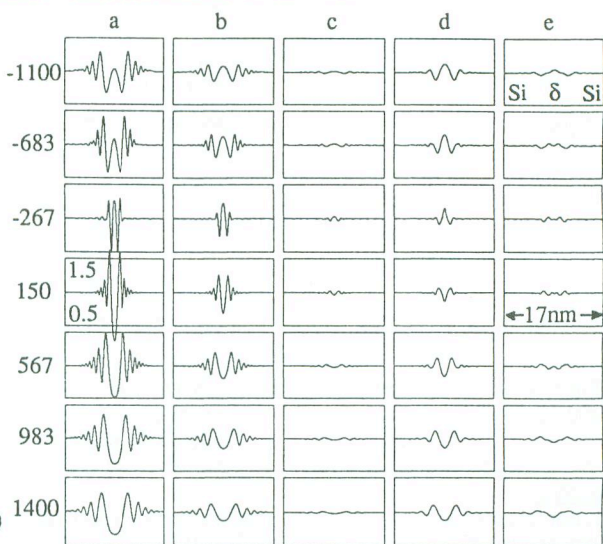


Fig.4 Simulations at the defoci shown (nm) and a crystal thickness of 100nm; a)100, b)24 and c)2.4at% boron in a single atomic layer; d) and e) show 24at% boron spread evenly from a sheet over 8 and 20 layers.

Retaining still the bulk silicon lattice parameter, we can next investigate the effect of 'diffuseness' on the contrast, incorporated here by spreading the boron in the form of a 'triangular' compositional profile. The profiles in Fig.6 show the effect of such triangular profiles with mean spreads of 20 and 16 atomic layers. It is clear that the latter profile is a better fit to the fringe spacing, and the contrast has the correct form as it approaches zero defocus even if it is still too high. Consequently we finally investigate the effect of changing the lattice parameter of the doped interlayer. Holloway and McCarthy (1993) have measured an experimental lattice contraction parameter for boron dopant in bulk silicon for lower boron contents, which we apply here by extrapolating their lattice parameter changes to the relevant higher B contents and by constraining the interlayer to match the lattice parameter of the silicon parallel to the interfaces, while retaining the predicted bulk volume. Examples of the contrast profiles then obtained are shown for an even compositional spread over 1 and 8 atomic layers in Figs. 7a and b respectively. Fascinatingly the predicted contrast has reversed for this full accommodation, and so no longer matches the sense of the experimental contrast. The explanation for this is clear when it is realised that the mean potential of boron in silicon when contracted to its experimental bulk volume is higher than that of silicon, and not lower as for any amount of boron on a substitutional lattice with silicon's planar spacing. Whatever the concentration of boron in our layer, it therefore cannot have

retained the volume it would have as a bulk dopant. The effect of changing the lattice parameter by a smaller amount is also shown in Fig.6. A change in the lattice parameter of the interlayer by only 0.2% for a 24at% sheet spread evenly over 20 atomic layers is dramatic - the contrast has gone down by 30%. Although the contrast could be matched with any profile by changing the lattice parameter of the interlayer appropriately, we must be restricted here to match the fringe spacing to a profile that is spread over at least 2nm and this is not possible using the extrapolated lattice parameter changes based on the data used above. Consequently it would appear that either the lattice parameter reductions associated with the incorporation of B at the levels present is lower than expected or that the B concentration present is less than the SIMS data suggest. The former possibility is being investigated using convergent beam measurements of the tetragonality and the latter by the use of HADF imaging.

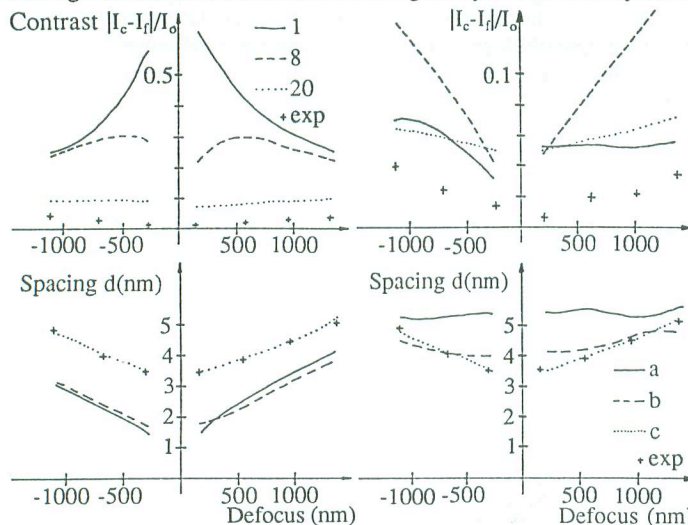


Fig.5 Simulated fringe contrast and spacing for 24at% boron (sheet density) spread evenly over 1, 8 and 20 atomic layers.

Fig.6 Diffuse profiles with mean spreads of a) 20 and b) 16 layers; c) interlayer plane spacing 0.2% ↓ for an even spread over 20 layers.

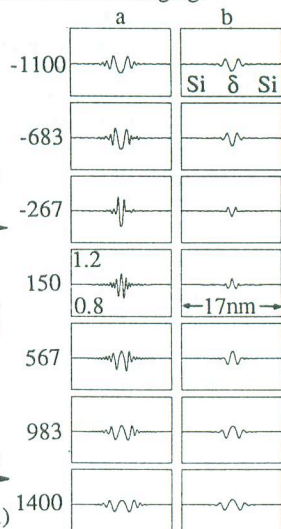


Fig.7 Even spread of B over a) 1 and b) 8 layers, retaining the predicted bulk volume.

#### In conclusion:

1. For this specimen, whatever the absolute boron concentration, it has diffused on average over about 2nm and has the form of a diffuse profile. If we believe that we started with a sheet density of 24at%, then the lattice must have contracted, but by much less than for the equivalent concentration in the bulk. In any case we here demonstrate that profiling concentrations of the order of 1at% boron or less is possible using the method. Significantly, the technique still works in the presence of absorption, which has not precluded an accurate characterisation.
2. In general, the Fresnel Method should be applicable to true, sub-nm delta doped layers with concentrations of 1at% or less.
3. In principle, the technique could therefore be used for measuring the diffusion coefficient of MBE-deposited boron in defect free material; measurements are normally made on implanted layers for which the diffusion is affected by implantation damage.

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