

# The determination of absorption parameters in Si and GaAs using energy filtered imaging

R E Dunin-Borkowski, R E Schäublin, T Walther, C B Boothroyd, A R Preston and W M Stobbs

Department of Materials Science and Metallurgy, Pembroke Street, Cambridge CB2 3QZ

**ABSTRACT:** Energy filtered imaging is applied to the determination of absorption parameters in Si and in GaAs through the comparison of bright and dark field two beam thickness fringes with computer simulations. The effect of energy filtering on the absorption parameters is discussed, and the accuracy to which the effects of specimen damage can be modelled using an effective Debye-Waller factor is assessed.

## 1. INTRODUCTION

A knowledge of accurate absorption parameters in electron diffraction is particularly important in the use of techniques such as quantitative convergent beam electron diffraction for the characterisation of electron densities. While theoretical predictions of contributions to absorption parameters have been determined (e.g. Bird and King (1990)), experimentally effects such as inelastic scattering and specimen damage are known to affect absorption parameters dramatically, as a function of the material examined and both the time for which the specimen is irradiated in the microscope and the objective aperture size used. Accordingly, here we investigate the effect of energy filtering on normal and anomalous absorption parameters in Si and in GaAs through the comparison of the intensities of thickness fringes with computer simulations.

## 2. EXPERIMENTAL DETAILS

Energy filtered images of GaAs and Si specimens were obtained at 400kV using a JEOL 4000FX microscope equipped with a post-column Gatan imaging filter (GIF). The use of a modified objective lens in this microscope, which has a further mini-lens fitted into its lower bore, allows reduced magnifications to be obtained prior to the spectrometer. The normal selected area aperture is then used as an objective aperture, the mini-lens being used to make this plane conjugate to the back focal plane of the objective lens. 90° cleaved wedge specimens of GaAs and Si were oriented to the Bragg condition for the 022 reflection, and the specimen tilts were refined by maximising the spacings of the thickness fringes visible in the images. The measured tilts from [100] were 8.3° and 6.5° for the GaAs and Si specimens respectively. Images were obtained at specimen temperatures of both 296K and 93K using a Gatan liquid nitrogen cooled double tilt holder. The objective aperture size used had a semi-angle of 2.06 mrad. (approximately half a Bragg angle,  $\theta_B$ ) and identical microscope lens settings were used for both specimens, with a beam convergence semi-angle of less than 0.05 mrad. Filtered images were obtained using a 6.4eV energy window, and a 3mm GIF entrance aperture was used to avoid scattering within the spectrometer (uniform backgrounds are added for larger apertures). All of the images were (512×512) pixels in size and were taken at exposure times of 1s, corresponding to an incident intensity of approximately 12,000

counts per channel. Sections of the experimental thickness fringes were projected accurately over a distance of 230nm using the Semper image processing software, and the point spread function of the GIF was deconvoluted (resulting in an increase in contrast of approximately 5% for the GaAs thickness fringes). The resulting one dimensional fringe profiles were aligned laterally and scaled to an incident intensity of unity. The variation in specimen thickness with distance in each profile was calibrated as 2.33nm/pixel. A high accuracy was achieved for this measurement by calibrating the magnification (maintained at a constant objective current) using several multilayers with known periodicities and weak beam thickness fringes at conditions insensitive to  $\xi_g$ . The data were consistent to 0.5%.

### 3. COMPARISONS WITH SIMULATIONS

The effects of absorption can be incorporated into simulation programs in many different ways. Here, we have used the 'Cufour' many-beam dynamical calculation of Schäublin and Stadelmann (1993), which obtains a solution of the Schrödinger equation by using a system of linear differential equations and the column approximation. Normal and anomalous absorption parameters are included as  $1/\xi_0'$  and  $\xi_g/\xi_g'$  respectively, where  $\xi_g$  is the extinction distance and  $\xi_0'$  and  $\xi_g'$  are normal and anomalous absorption distances respectively. Simulated thickness fringe profiles were matched to both bright and dark field experimental profiles at each imaging condition by varying the values of Debye-Waller factor (DWF), normal and anomalous absorption coefficients, and misorientation from the exact Bragg orientation. Values of  $\xi_g$  were taken from the calculations of Weickenmeier and Kohl (1991) and were not varied, but are not expected to be in error by more than 1-2%. The difference between the experimental and simulated images was minimised to give best-fitting profiles whose parameters are listed in table 1. A selection of experimental and fitted thickness fringe profiles is also shown in figs. 1a and 1b for unfiltered and filtered 93K data respectively.

Material	Filtering	Temp. (K)	$1/\xi_0'$ (nm <sup>-1</sup> )	$\xi_g/\xi_g'$	DWF (nm <sup>2</sup> )	Tilt ( $\theta_B$ )
GaAs	Unfiltered	93	0.0011	0.07	0.006	0.025
GaAs	Unfiltered	296	0.0011	0.07	0.010	0.025
GaAs	Filtered	93	0.0018	0.07	0.008	0.025
GaAs	Filtered	296	0.0018	0.07	0.010	0.025
Si	Unfiltered	93	0.0004	0.02	0.014	0.05
Si	Unfiltered	296	0.0004	0.002	0.017	0.05
Si	Filtered	93	0.0010	0.03	0.014	0.05
Si	Filtered	296	0.0010	0.002	0.017	0.05

Table 1. Best-fitting parameters to the experimental data.

The following observations are apparent from table 1 and fig.1:

1. A surprising feature of the experimental data is that the minima in the filtered low temperature fringes are not lower. In table 1, this effect is fitted by using a combination of a slight misorientation from the Bragg condition and a non-zero value of anomalous absorption  $\xi_g/\xi_g'$ . Although the sense of the difference in  $\xi_g/\xi_g'$  between GaAs and Si is consistent with Si having a higher Debye temperature  $\theta_D$  (645K) than GaAs (360K), it is disturbing to note that Si appears to have a higher value of  $\xi_g/\xi_g'$  at the lower specimen temperature.
2. Energy filtering is seen to radically change the best fitted values of the normal absorption coefficients  $1/\xi_0'$  for both materials.
3. Although an increase in specimen temperature increases the fitted value of the DWF in both materials as would be predicted, it is surprising that the fitted values are all much larger than those which are typically quoted for temperatures of 93K and 296K (i.e. 0.0025 and 0.0066nm<sup>2</sup> for GaAs and 0.0022 and 0.0046nm<sup>2</sup> for Si). Fig.1c shows simulations which incorporate such quoted values for a temperature of 93K, and these clearly have fringe spacings which are inconsistent with the experimental data. (It should be noted that the DWF is the only major parameter, apart from specimen tilt, which

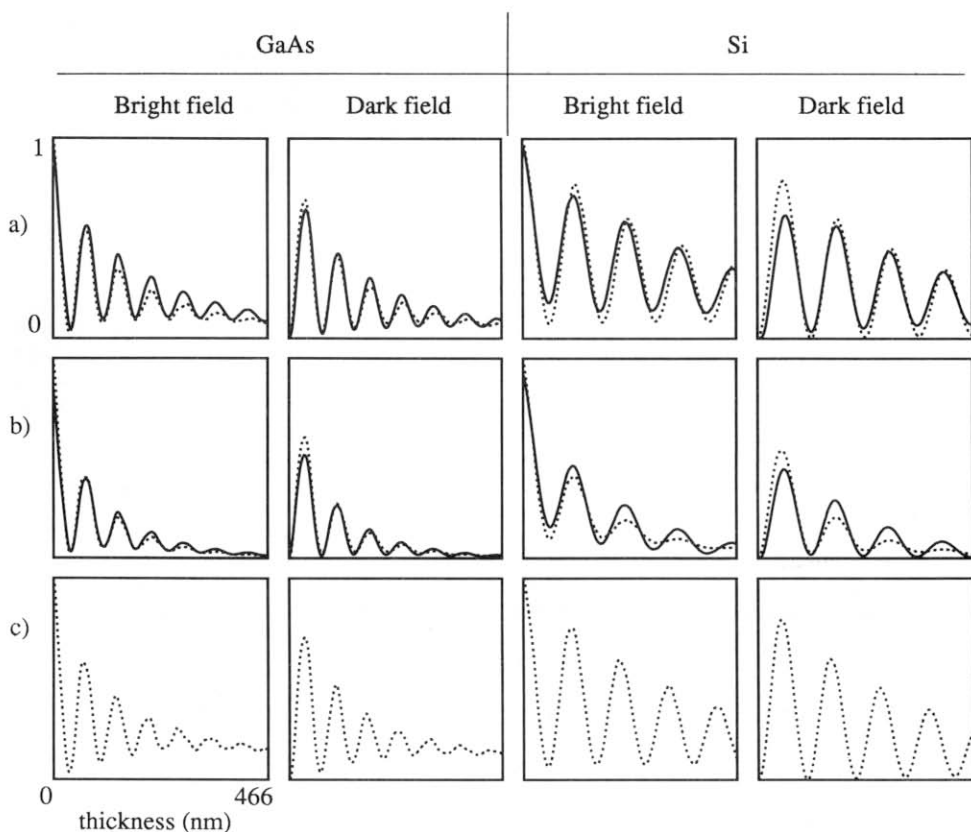


Fig.1 a) Unfiltered and b) energy filtered small objective aperture 93K experimental data (solid lines) and best-fitting computer simulations (dotted lines) as described in table 1. The simulations in c) are as for a) but with DWFs of 0.0025 and 0.0022nm<sup>2</sup> for GaAs and Si respectively.

controls the fringe spacing for a fixed value of  $\xi_g$ , and that such an effect would not have been visible in an ion-thinned specimen for which the specimen geometry were not known).

4. The simulations do not fit the data perfectly, and this is particularly clear for the first bright fringe in the dark field images which is consistently lower experimentally than in the simulations.
5. While the spacing of the GaAs thickness fringes is constant with thickness, the Si fringe spacing increases with thickness at a specimen temperature of 93K and decreases with specimen thickness at 296K, as shown in fig.2. The same area of the specimen was examined at the two temperatures, and there were no dynamical beating effects in the thickness fringes.

#### 4. DISCUSSION AND CONCLUSIONS

The most likely explanation for the surprisingly large DWF required to fit the data lies in the fact that the images which were examined here are taken from longer series of images, for which each specimen was examined for a total of several hours at 400kV. The observed features are thus probably related to electron beam-induced damage. Hall et al. (1966) have

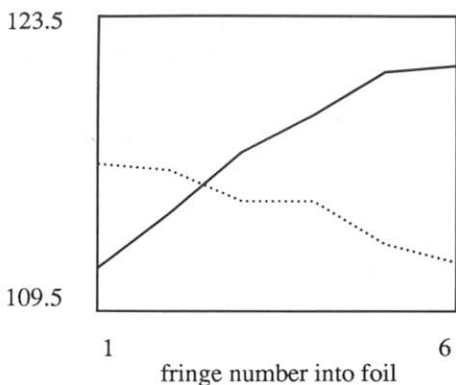


Fig.2. Experimental thickness fringe spacing (nm) for Si plotted as a function of distance into the specimen for a specimen temperature of 93K (solid line) and 296K (dotted line).

and the dependence of the form of the knock-on damage.

The fact that in all aspects other than the fringe spacing the profiles in fig.1c provide a better fit to the experimental data than the best-fitting simulations indicates that the use of an effective DWF does not model the effects of specimen damage accurately. The presence of an amorphous layer on the specimen surfaces has also not been considered here, and may require normal absorption to be modelled using two exponential decays rather than one (Dobson et al., 1991) and this would phenomenologically allow a better contrast fit for the first thickness fringe.

A further observation is that in the experimental data for GaAs in fig.1 the *unfiltered* image has thickness fringes of large amplitude at high thicknesses, suggesting that the contrast of plasmon loss electrons is high and hence that they add to the contrast of the elastic contribution (see also Walther et al. (1995)).

Our conclusions are as follows:

1. There is a large difference between the value of the normal absorption parameter  $1/\xi_0'$  in filtered and unfiltered thickness fringe data, and the change is qualitatively as expected.
2. Careful examination of the data suggests that the presence of electron beam-induced damage changes both the qualitative appearance of the thickness fringes and the values of the fitted absorption parameters. In particular, the use of an anomalously large value of the DWF is required to match the experimental fringe spacings.
3. Such an increase in the effective DWF does not result in a good fit of the absorption parameters to the experimental fringe intensities, and is probably not a good model for the effects of specimen damage.
4. The coherence of plasmon scattered contributions is suggested by the amplitude of the thickness fringes in unfiltered data at high specimen thicknesses.

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stated that such effects can produce an effective increase in the "DWF" of the material, and such an increase is observed for both specimens. Indeed, it is experimentally more pronounced for Si which is known to damage faster than GaAs. (The high resolution images of Walther et al. (1995) are not consistent with such an increase in DWF, however their specimen was examined for a much shorter time). This explanation is also consistent both with the observed finite value of  $\xi_g/\xi_g'$  in the low temperature filtered data and with the larger value of  $\xi_g/\xi_g'$  for Si at low temperature, as these images were taken *after* the high temperature data. The change in the spacing of the Si fringes with thickness suggests that there is a change in the "effective DWF" associated with specimen damage as a function of thickness and temperature. This might be explained by the effect of variations in the interstitial depletion depth