The Quantitative Treatment of Errors when using Fourier Coefficients of HREM Image Contrast to Profile the Composition of GeSi

R. E. Dunin - Borkowski and W.M. Stobbs

Department of Materials Science and Metallurgy, Cambridge University, Pembroke Street, Cambridge CB2 3QZ, U.K.

1. INTRODUCTION

Several authors have investigated the sensitivity of HREM image contrast to changes in composition for coherent $Ge_{1-x}Si_x$ interfaces. Notably, Stenkamp and Jäger (1993) showed that at 400kV the amplitudes of the 220 and 111 Fourier coefficients at the <001> and <01$\bar{1}$> zone axes respectively can have a quasi-linear dependence on x in optimised thickness-defocus ranges. It was stated both that strain and Fresnel contrast have a negligible effect on the coefficients and that the contrast can be compared directly with that of 'bulk' GeSi. At the <001> zone axis optimum thickness and defocus ranges of 0 to 18nm and -60 to -50nm respectively were determined for a JEOL 4000EXII. The amplitude of the 220 coefficient was found to vary monotonically with x, and an accuracy of better than 0.1 in x was quoted for fitted compositional profiles. However, the sensitivity to composition over that due to strain was not *quantified*. Also, one disadvantage of the technique is that in an experimental image the compositions of two separate regions that exhibit different contrast must be known before a graph of the relationship between the Fourier coefficient and the compositon can be established.

2. CALCULATIONS

Our aims here are firstly to develop a method for quantifying the sensitivity to composition over that due to strain, and secondly to establish whether this HREM method can be applied if only one reference composition is known. To illustrate the approach, the specific case of the characterisation of GeSi layers of unknown concentration grown on a pure Ge substrate will be simulated, as at <001> using an objective aperture of semi-angle 20mrad (Airy disc radius 0.050nm) and JEOL 4000EXII parameters. It will be assumed that experimental parameters such as specimen thickness, defocus, beam convergence and focal spread are known to high accuracy and that contributions to the contrast from contamination layers, inelastic scattering, microscope misalignments as well as Fresnel effects are negligible! Strained lattice parameters included in multislice simulations are calculated using the method of Zunger and Wood (1989).

We firstly consider the quantitative assessment of the effect of *strain* on the measurement of compositions (*even* if two reference values are known). Fig.1 shows as a function of specimen thickness and for a defocus of -55nm the maximum possible error in the composition of GeSi grown on a Ge substrate measured using the amplitude of the 220 Fourier coefficient at <100>. This error is caused by assuming that contrast from a *strained* region is the same as that from an unstrained region of the same composition. Not surprisingly, the largest error in the measured composition on pure Ge is for Si-rich layers. For confidence in the composition to better than 10% for all possible compositions the thicknessmust be greater than 12nm (and *not* 0-18nm).

Non-linearity of the amplitude of the Fourier coefficient with composition and the problem of using only one reference point will now be considered. Typical graphs of the amplitude of the 220 coefficient as a function of *composition* are shown in Fig.2 for a defocus of -55nm and

at the thicknesses indicated (nm). There is a clear change in linearity with specimen thickness. The form (although not the magnitude) of the composition can only be determined *without* the use of two reference compositions if a range in thickness can be found for which the coefficient is linear with composition for all compositions. Accordingly, Fig.3 shows the maximum error in the measured composition of GeSi grown on a Ge substrate caused by non-linearity of the coefficient with composition, for defoci of -50, -55 and -60nm (ignoring the effect of strain described above). The acceptable thickness range at each defocus for the error in the measured composition to be less than 10% is very narrow. The shift of this range to a different thickness with defocus is surprisingly rapid and suggests that even if the optimum specimen thickness at a given defocus can be chosen, the defocus itself must be known to better than a few nm.

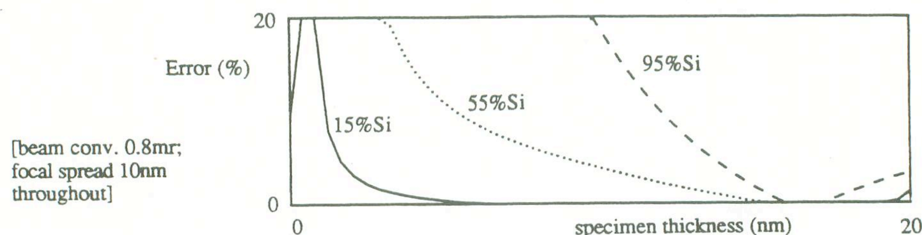

Fig.1 The maximum error in the measured composition of GeSi on Ge, caused by assuming that strained and unstrained regions of the same composition have identical contrast.

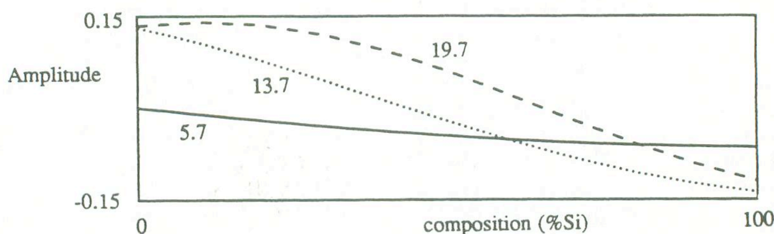

Fig.2 Amplitude of the 220 Fourier coefficient vs composition at a defocus of -55nm and at the thicknesses indicated (nm).

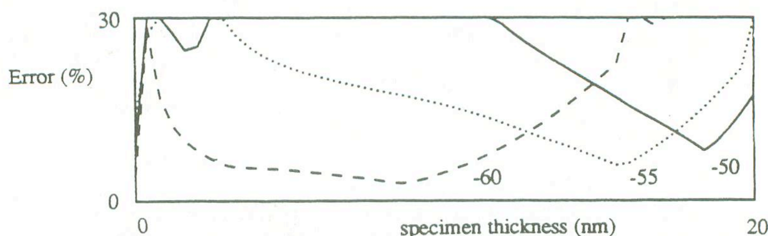

Fig.3 The maximum error in the measured composition of GeSi on Ge caused by non-linearity of the coefficient with composition, for the defoci indicated (nm).

3. CONCLUSION

Methods for the quantitative assessment of the accuracy of composition measurements in GeSi using Fourier coefficients of HREM image contrast have been presented. It has been shown that the thickness and defocus ranges over which the technique can be applied to high accuracy are narrower than previously thought, especially if only one reference composition is known.

Stenkamp D. and Jäger W. (1993), Ultramicr. **50**, 321
Zunger A. and Wood. D.M. (1989), J. Cryst. Growth **98**, 1