Analysis of partially oxidised epitaxial silicon mono-layers on germanium virtual substrates using aberration corrected scanning transmission electron microscopy

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Abstract: We report the application of aberration corrected STEM imaging to study the interface structure of a partially oxidised epitaxial-Si passivation layer, nominally 2 mono-layers thick, deposited on a Si(001)/Ge virtual substrate. HAADF imaging revealed evidence of 1-2 mono-layers of crystalline Si between the Ge virtual substrate and overlying SiO₂. An interface roughness of 1-2 mono-layer steps was also apparent between the epitaxial-Si and Ge virtual substrate. The total thickness of the residual crystalline Si and overlying SiO₂ suggests that the initial epitaxial-Si layer may have been 1-2 mono-layers thicker than the nominal value.

1. Introduction

Germanium on silicon based metal oxide semiconductor (MOS) field effect transistors offer potentially improved performance due to an enhanced carrier mobility compared to conventional silicon based technology [1]. One drawback of the application of pure germanium in MOS devices is the poor processing characteristics of GeO₂ compared to SiO₂ [1,2]. Hence, a critical step in the development of such devices is the deposition of a few mono-layers (ML) of partially oxidised epitaxial silicon (epi-Si) passivation between the germanium virtual substrate (Ge-VS) and a high-k hafnium oxide dielectric cap. The thickness and quality of this Si layer has a significant impact on the ultimate device performance [3] therefore knowledge of its structure and nature of the interface with the Ge-VS is desirable. In previous studies [1,4] the spatial resolution offered by conventional transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) has been insufficient to clearly elucidate the detailed structure of the interface. Hence in the current study, we have applied aberration corrected STEM imaging to investigate the microstructure of a partially oxidised epi-Si layer with a nominal thickness of half a unit cell deposited on pure Ge-VS. Analysis was performed in a 300kV aberration corrected TEM/STEM equipped with a cold-field emission gun and both probe and image correction [5]. The high angle annular dark-field (HAADF) and bright-field (BF) STEM resolution provided by this instrument is routinely better than 1Å. Moreover, the chemical composition sensitivity of the atomic number (Z) contrast offered by HAADF imaging can in principle reveal information about the nature of the Ge-VS/Si interface and the degree of oxidation of the epi-Si layer.

2. Experimental

Device layers were prepared on a ~2µm relaxed germanium virtual substrate deposited on (001) Si wafers using conventional silicon processing techniques at 650°C. Following a short H₂ anneal, an epi-Si layer was grown at 500°C using SiH₄. By adjustment of the process time and temperature
the thickness of the epi-Si layers can be controlled, in this instance to nominal ~2ML. Partial oxidation was achieved by subsequent immersion in 1ppm ozone for 60 seconds before deposition of an 8nm thick HfO$_2$ high-k dielectric cap by atomic layer deposition. Full details of the device processing route are provided elsewhere [1].

Cross-sections for examination in the TEM/STEM were prepared along the [110] direction by conventional mechanical polishing and thinning to electron transparency using Ar$^+$ ion beam milling. Imaging was performed in a 300kV cold field emission gun (C-FEG) double aberration corrected TEM/STEM (JEOL Z3100-R005). STEM imaging was performed with both the imaging and probe forming corrector optics optimised to provide a Cs value in each case less than ±1μm. Under such conditions transfer beyond 0.1nm is routinely achieved [5]. High angle annular dark-field (HAADF) STEM images were recorded with a probe current of 9.4pA, a convergence semi-angle of 21mrad and a STEM dark-field inner and outer detection angles of 62mrad and 165mrad respectively. Bright-field (BF) STEM images were collected using a Gatan BF detector at a nominal camera length of 12cm. Specimen thickness was estimated by analysis of the zero energy-loss peak using the standard routine within the Gatan Digital Micrograph (DM) software.

To assess the relative atomic column intensities in the HAADF images subtraction of the underlying background signal was performed. Firstly, the image intensity recorded in vacuum was subtracted from the raw image followed by a light 3x3 band-pass filter to reduce detector noise. The underlying background signal was then removed by assuming the background intensity between each row of atomic columns within the HAADF image was the same as that under the atomic columns. A series of contrast intensity profiles were subsequently generated for each row of atomic columns using the Gatan DM software each integrated over three pixels [6]. The contrast profiles were finally normalised with respect to the Ge columns to give a series of contrast ratio profiles. Similar analysis was extended to regions of the specimen of known composition, i.e. the interface between the pure Si (wafer substrate) and pure Ge (virtual substrate) to provide an internal calibration of the effective HAADF contrast.

3. Results and Discussion

A low magnification BF-STEM image, Fig. 1a, provides an overview of the device structure with the main regions of interest labelled. Fig. 1b shows a BF-STEM image of the interface between the (001) Si substrate and the overlying Ge-VS. A periodic modulation in the image contrast is observed at the interface with a period of ~10nm. This periodicity corresponds to complete relaxation of the lattice mismatch of the Si and Ge ($a_{Si}=0.5431$ nm and $a_{Ge}=0.56575$ nm) where $25x \Delta Ge_{(110)} \sim = 26x \Delta Si_{(110)} = 9.97$ nm. Closer examination of the regions indicated in Fig. 1b reveals a series of stacking faults just above the Si/Ge-VS interface, an example of which is shown in the HAADF image, Fig. 1c.

![Fig. 1](image-url)

Fig. 1: (a) BF-STEM overview of the bulk device architecture, (b) BF-STEM image of the interface between the (001) Si substrate and the Ge-VS illustrating the periodic contrast variations at the interface (arrowed), (c) HAADF image of one such region indicated in Fig. 1b revealing stacking faults (bound by white arrows).
Fig. 2: (a) HAADF STEM image of the Ge-VS/Epi-Si interface (growth direction left to right) the 0.14nm germanium dumbbells are clearly resolved within the Ge-VS region, (b) the corresponding BF STEM image and (c) contrast intensity profiles across adjacent rows (i) and (ii) of atomic columns defined in 2(a).

Fig. 3: Plot of theoretical HAADF SiGe/Si Z-contrast dependency for a range of Si-Ge binary alloys ($Z^2$, $Z^{1.7}$ and $Z^{1.4}$) and experimentally derived effective contrast intensity ratio for pure Ge and Si (Ge/Si=3.25±0.23).

Fig. 4: Composite HAADF and BF image with the atomic columns designated as either Ge or Si based on the relative $I_{Ge}/I_{Si}$ contrast ratio measured from each row of atomic columns as illustrated in Fig. 2c.

Fig. 2a and 2b show a HAADF and corresponding BF STEM image respectively of the surface Ge-VS/Si/SiO$_2$/HfO$_2$ interface region (from left to right), the Ge (004) dumbbells (0.14nm) are clearly resolved in the virtual substrate and the HfO$_2$ appears with bright contrast to the right hand side of Fig. 2a. The atomic number contrast offered by HAADF imaging provides a means of extracting information about the atomic species through measurement of the relative image contrast. Fig. 2c shows two contrast intensity profiles, (i) and (ii), from adjacent rows of atomic columns after back ground subtraction and normalisation. Both profiles indicate a reduction in the relative intensity of the atomic column contrast at the interface between the Ge-VS and the amorphous oxide speculatively labelled as crystalline Si (c-Si) in Fig. 2c. The mean experimental contrast intensity ratio derived from Fig. 2a ($I_{Ge}/I_x$) (where $x$ corresponds to the intensity of the suspected Si atomic columns) corresponds to 2.75±0.30 which is significantly lower than the expected of ~5.2 assuming a $Z^2$ dependency predicted by pure Rutherford scattering. A plot of the
Theoretical contrast intensity ratios for the binary Si:Ge system for a range of $Z$ dependencies is presented in Fig. 3. In order to assess the dependency observed in our current data, we have performed an internal calibration for regions of known composition (pure Si substrate and pure Ge-VS). The experimental results suggest an $I_{Ge}/I_{Si}$ contrast ratio of 3.25±0.23, i.e. closer to a $Z^{1.4}$ dependency as indicated in Fig. 3. Within the experimental error this value approaches those observed for the profiles obtained in Fig. 2c and provides some confidence that 1-2 ML coverage of c-Si remains after partial oxidation. Subsequently, by similar analysis of the $(I_{Ge}/I_{x})$ profiles for each row of atomic columns in Fig. 2a we are able to assign atomic columns with a $(I_{Ge}/I_{x})$ ratio between 2.4-3.5 as Si and those with ratios approaching unity as Ge as illustrated in Fig. 4. A transition region with values between these two extremes is evident suggesting apparent atomic column intermixing within the transition region and/or an interface roughness of 1-ML extending along the electron beam direction through the sample. Alternatively, delocalisation of the probe intensity to neighbouring atomic columns through the relatively thick sample, determined as ~33nm by energy-loss spectroscopy may also be significant [7]. Factors including detection angle and specimen thickness can all potentially influence the relative HAADF contrast while the presence of interface strain in similar structures has been shown previously to modulate the $Z$-contrast under near zone axis conditions [8]. The thickness of the overlying SiO$_2$ layer was estimated at 0.60-0.68nm. The interface between the SiO$_2$ and HfO$_2$ was taken as the half maximum of the contrast intensity gradient however the exact nature of this interface was difficult to assess. Future spectroscopic profiling and image simulation are therefore required to support the experimental STEM data before further conclusions can be drawn.

4. Conclusions

Partially oxidised epitaxial-Si passivation layers, nominally 2 ML thick, deposited on pure Ge-VS have been investigated using aberration corrected STEM BF and HAADF imaging. Studies of the (001)-Si/Ge-VS interface reveal the presence of periodic stacking faults originating several ML above the interface. Analysis of the HAADF $Z$-contrast at the Ge-VS/epi-Si interface indicates that 1-2 ML of c-Si remains after surface oxidation. In addition, an apparent projected interface roughness between the Ge VS and c-Si region of 1-2 ML is observed. The total thickness of the remaining c-Si and overlying SiO$_2$ suggests that the initial epi-Si layer must have been 1-2 ML thicker than the nominal value. The nature of the interface between the amorphous SiO$_2$ and the HfO$_2$ dielectric cap however is less distinct and requires further clarification.

Acknowledgements

The authors would like to thank the Engineering and Physical Sciences Research Council for their financial support (EP/F033893/1 – “Renaissance Germanium”).

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