Evaluation of the substitutional carbon content in annealed Si/SiGeC superlattices by dark-field electron holography

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Abstract. Si/SiGeC superlattices can be used as core structure for the fabrication of new microelectronic architectures with enhanced electrical properties such as multichannel transistors. The introduction of carbon in SiGe allows for compensation of the compressive strain and to avoid plastic relaxation. However, the formation of incoherent β-SiC clusters during annealing at high temperature limits the processability of SiGeC. This precipitation leads to a strong modification of the stress in the alloy due to the reduction of the substitutional carbon content. Here, we investigated the variation of the substitutional C content in annealed Si/SiGeC superlattices using a comparison of dark-field electron holography and finite element simulations.

1. Introduction
Multichannel devices have recently been proposed as an alternative architecture of metal oxide semiconductor field effect transistors (MOSFETs) for future microelectronic technologies [1]. They offer a high current drivability and have a relative immunity to short-channel effects. Their fabrication relies first on the epitaxy of Si/SiGe superlattices (SLs) [2]. After deposition on silicon-on-insulator substrate, the SLs are patterned to define the source and drain regions. The SiGe layers are then selectively etched and the voids left between the suspended Si beams are filled with a gate stack.

Alloying the SiGe layers with carbon is interesting because it allows the germanium concentration and the strain in the layers to be independently controlled. A high germanium content can be used while maintaining a low level of strain with a small amount of carbon in substitutional position. However, during annealing at high temperature (> 900°C), the strain behaviour of SiGeC can be modified by the formation of β-SiC clusters [3]. These clusters are incoherent with the surrounding matrix [4] and the C atoms incorporated are therefore lost from the point of view of the global strain distribution. Several characterization techniques can be used to monitor the reduction of the substitutional C content in SiGeC during thermal processing. Most common are Fourier transformed infrared spectrometry (FTIR), X-ray diffraction (XRD), Rutherford and resonant backscattering spectroscopy (RBS/rBS). However, all of them have a relatively poor lateral resolution. Here, we propose a method that could be applied to nanometric structures. It consists of a combination of a recent transmission electron microscopy (TEM) technique: dark-field electron holography (DFEH) [5] with finite element simulation (FEM). DFEH has been shown to open new possibilities for the
measurement of strain in semiconductor devices. It provides a large field-of-view (up to 1x0.25 µm²) and a spatial resolution of 10 to 4 nm [5]. Moreover, the comparison with simulations is direct which allows structural and compositional information to be rapidly linked.

2. Experimental procedure

The Si/Si₁₋₀.₇₄₄Ge₀.₂₄₄C₀.₀₁₂ superlattices (SLs) consist of four periods epitaxially grown by reduced pressure chemical vapour deposition (RP-CVD) on <100> Si wafers [2]. Intrinsic Si/Si₁₋₀.₇₈₆Ge₀.₂₁₄ SLs were also created and were used as a reference for simulations. The growth was conducted in an Epi Centura cluster tool from Applied Materials at 650°C and 20 Torr. The gaseous precursors were SiH₂Cl₂ and GeH₄ for the SiGe layers and SiH₄ for the Si layers. SiCH₆ was added for the SiGeC layers. Samples were then annealed in H₂ for two minutes at different temperatures. The following conditions are described in this paper: as deposited and annealed at 1050°C for the Si/SiGe SLs; as deposited, annealed at 950°C and 1050°C for the Si/SiGeC SLs. The distribution of Ge and C was determined by time of flight secondary ion mass spectrometry (ToF-SIMS) using an ION-TOF TOF SIMS V instrument. Ge depth profiles are shown in figure 1.

Dark-field electron holography was performed on a FEI Titan transmission electron microscope with an operating voltage of 200 kV. The objective lens was switched off and the holograms were acquired using a Lorentz lens. The biprism was biased with a voltage of 180 V corresponding to a fringe spacing of 2 nm. Information in the growth direction was obtained by selecting the (004) diffraction spot with a 10 µm objective aperture. Acquisitions were performed during 64 s for signal-to-noise improvement. More details about the experimental conditions and post-treatment method are described in Béché et al. [6]. Samples were prepared into [110] parallel-sided cross sections using a dual beam FEI Strata 400 with a final milling step at 8 kV.

Finite element simulations of the strain in the lamellas were carried out using the structural mechanics module of COMSOL Multiphysics. The plane strain approximation was made to simulate the samples in 2D and save computing time. The lamella geometry was constructed, taking into account the crystalline thickness, determined by convergent beam electron diffraction (CBED) and the dimensions of the layers measured by X-ray reflectivity. The SiGe and SiGeC alloys stiffness coefficients were calculated according to Vegard’s law. The SiGe lattice parameter a_{SiGe} was estimated using an empirical law based on Dismukes’s results [7]:

\[ a_{Si_{1-x}Ge_x} = a_{Si} + 0.2x + 0.026x^2 \] (1)

The SiGeC lattice parameter a_{SiGeC} was calculated according to a law proposed by De Salvador et al. [8]:

\[ a_{Si_{1-x-y}Ge_xC_y} = (1 - x - y)a_{Si} + xa_{Ge} + ya_{C} - 0.026x(1 - x) - 0.59y(1 - y) + 0.06xy \] (2)

where \( a_{E} \) is the lattice parameter of the element E, x and y are respectively the Ge and C contents. The concentrations were taken directly from the ToF-SIMS depth profiles using a MATLAB program. Only substitutional C atoms were taken into account in the model. It was supposed that non-substitutional C atoms have a negligible influence on the strain state [8]. Several simulations were performed for different substitutional content until a good fit was obtained with the experiment.

3. Results

3.1. Dark-field electron holography and ToF-SIMS profiling

Strain maps reconstructed from dark-field electron holograms are shown in figure 2. Strain profiles extracted from the maps in the growth direction are represented as continuous lines in figure 3. In the case of the intrinsic Si/SiGe SL (figure 3(a-b)), the distribution of the strain becomes smoother and broader after annealing at 1050°C which is due to the germanium interdiffusion (figure 1(a)). It induces a decrease of the maximum strain level in the SiGe layers from 1.2 to 0.9%.

Figure 3(c) shows the strain profiles corresponding to the Si/SiGeC superlattices. For the as-grown sample, the strain level in the SiGeC layers is equal to 0.9% which is less than the value measured in
the intrinsic superlattice (1.2%). Whereas the germanium content is higher in the C doped layers (24% against 21% in the intrinsic layers), the strain level is lower, meaning that it is effectively reduced by the presence of carbon. The diffusion of the germanium is more important in these samples (figure 1(b)) than in the intrinsic ones. This comes from the presence of the carbon which strongly reduces the activation energy of the Si-Ge diffusion process. Although the strain distribution is broader at 1050°C than at 950°C, the level of the strain is also slightly higher. It indicates that the strain compensation induced by the substitutional C atoms is reduced, which is linked to the formation of β-SiC clusters.

Figure 1. Ge ToF-SIMS concentration profiles for the (a) Si/SiGe and (b) Si/SiGeC superlattices.

Figure 2. (a) Example of dark-field electron hologram. (b-f) Reconstructed strain maps of samples: (b) Si/SiGe as deposited, (c) Si/SiGe annealed at 1050°C, (d) Si/SiGeC as deposited, (e) Si/SiGeC annealed at 950°C and (f) Si/SiGeC annealed at 1050°C.

Figure 3. Average experimental and simulated strain profiles in and along the [001] growth direction of samples: (a) Si/SiGe as deposited and (b) Si/SiGe annealed at 1050°C. (c) Si/SiGeC experimental strain profiles. (d-e-f) Simulations for different substitutional C contents, compared to the experiment.

3.2. Finite element simulations
The simulations corresponding to the Si/SiGe superlattices are shown as dashed lines in figure 3(a-b). Globally, the experiment was found to be in excellent agreement with the simulated strain profiles, for both the as-grown and annealed SiGe samples. The strain in the uppermost layer is however slightly
lower than in the other layers especially in the case of the annealed sample which is not confirmed by the simulation. It can be linked to a variation of the foil thickness close to the edge of the specimen.

Simulations corresponding to the C doped samples and for three different substitutional C contents are represented in figure 3(d-e-f). In the case of the as-grown sample (d), the experimental profile is in accordance with the simulation for a substitutional C content of 0.75% ± 0.05%. Compared to the total C concentration of 1.24%, it indicates that 61% ± 4% of the C content is in substitutional position. Similarly, at 950°C, the agreement between experiment and simulation was obtained for a substitutional C content of 0.60% ± 0.05% (or 48% ± 4% of the total C concentration). For the sample annealed at 1050°C, the match was obtained for 0% of substitutional C. We note that when inputting y = 0 in the equation (2), it leads to the Dismukes expression (1) which is known to be valid for intrinsic SiGe. It shows that from the point of view of strain, the sample behaves like pure SiGe.

4. Discussion
Relying on the conclusions deduced from the comparison of DFEH and simulations, the behaviour of the Si/Si$_{0.744}$Ge$_{0.244}$C$_{0.012}$ superlattice during annealing can be explained in greater detail. It was noticed that although there is an important diffusion of the germanium, the level of the strain is slightly higher at 1050°C than at 950°C. This can be explained by the massive decrease of the substitutional C content for the highest annealing temperature. At 950°C, there is still 48% of the total C content in substitutional position (compared to 61% for the as-grown sample) and the strain in the superlattice is reduced due to the strong interdiffusion of the germanium. On the contrary, at 1050°C, there is no more substitutional C and the related strain compensation is therefore lost, allowing the strain level to reincrease. However, because this phenomenon is in competition with the diffusion of the germanium, the reincrease is not so important and the effective strain level is almost the same at 950°C and 1050°C.

5. Conclusion
The influence of thermal processing at high temperature on the strain in Si/SiGeC superlattices has been studied by dark-field electron holography. The strain behaviour of the SL was found to be coherent with the formation of SiC clusters. Finite element modelling was then used to simulate the distribution of the strain in the TEM lamellas. As a good correlation was obtained between experiment and simulation for intrinsic Si/SiGe superlattices, the same method was applied to the investigation of the Si/SiGeC SLs. It was found that the most important loss of substitutional C occurs at 1050°C and it eliminates the effect of C on the strain. At this temperature, the strain is governed essentially by the distribution of the germanium which is highly diffused in comparison with the intrinsic superlattice.

This study demonstrates the possibilities offered by the combination of DFEH strain mapping and finite element simulations for linking structural and compositional information at the nm-scale. The method could be applied to the investigation of the substitutional C content in SiGeC recessed source and drain transistors for example. This would help for the understanding of the strain level in the channel and therefore the electric properties of the devices.

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