STRUCTURAL CHARACTERIZATION OF SiGe AND SiGe:C HETEROSTRUCTURES USING A COMBINATION OF X-RAY SCATTERING METHODS

J.F. Woitok
PANalytical B.V., PO Box 13, NL-7600 AA Almelo, The Netherlands

ABSTRACT

This study is about the structural properties of SiGe and SiGe:C heteroepitaxial layers on Si (001). The structural characterization is based on the application of complementary information content of X-ray scattering techniques like high-resolution X-ray diffraction (XRD), X-ray reflectivity (XRR) and X-ray diffuse scattering (XDS). One main focus of the analysis is to derive a sample model that sufficiently describes all experimental data sets. In addition, the reliability of parameters extracted by just one single method is discussed. It turned out that XRR is more sensitive to the near surface region indicating the presence of surface roughness and density gradients that do not significantly affect the XRD pattern.

INTRODUCTION

In the rapidly growing market for high performance radio-frequency devices Silicon-Germanium (SiGe) has demonstrated that it plays a dominant role [1]. The importance of the SiGe technology is well known for applications that require high-speed performance at reduced power consumption, such as cell phones. Key properties of these devices, such as maximum operating frequency, are very sensitive to process variations during epitaxial growth which can lead to slight changes in layer composition and thickness. In the recent generation of SiGe-based heterojunction bipolar transistors (HBTs) Carbon has been incorporated in order to reduce the strain and to reduce the diffusion of dopants like Boron in base region [2]. This imposes additional challenges on characterization tools used to determinate accurately thickness and chemical composition. High resolution X-ray diffraction (XRD) is nowadays a well-established technique for the non-destructive ex-situ investigation of Si-based epitaxial heterostructures [3]. It non-destructively provides absolute, calibration free, precise information about composition, strain and thickness of semiconductor hetero-epitaxial layers. However, in order to obtain a full structural characterization; the complementary information content of high-resolution X-ray diffraction, X-ray reflectivity (XRR) and X-ray diffuse scattering (XDS) should be applied [4, 5]. The latter techniques allow the extraction of quantitative information about thickness, interface layers, vertical interface roughness and lateral correlation length regardless the crystallinity of the materials involved. One important advantage of the combination of scattering methods is the independent determination of thickness values. The basis of the analysis of all scattering data is a sample model considering the growth sequence. Subsequently the corresponding layer parameters are refined until a close match of full pattern simulations to the measured data is achieved. This last step, originally a time-consuming trial and error procedure, is nowadays significantly simplified by the availability of fast and reliable automatic refinement algorithms [6, 7]. All of the steps in the present study were performed by means of commercially available software and hardware.
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products [8]. The versatile concept of modern laboratory X-ray equipment easily allows all type of experiments to be performed on a single instrument just by exchanging optical modules without time-consuming alignment steps.

**EXPERIMENTAL**

SiGe heterostructures grown by reduced pressure chemical vapor deposition (RP-CVD) from different sources were studied. All structures were grown on (001) Si substrates. Single layers, typical Heterojunction Bipolar Transistor (HBT) structures, step graded as well as linear graded SiGe structures capped with Si layers were analyzed. A typical sample size was several cm$^2$. Additionally SiGe:C layers on 8 inch Si were evaluated. Two typical examples of the different kinds of structures were chosen to demonstrate the capability of the combination of scattering techniques. All measurements were performed on a PANalytical X"Pert PRO MRD [9]. Cu radiation and a four-crystal Ge monochromator in the (220) setting were applied for the high-resolution diffraction scans. The beam size was limited to 1.4 x 2.5 mm$^2$. All of the samples had $\omega - 2\theta$ scans measured around the symmetrical (004) reflection with a 1 x10 mm$^2$ slit in front of the detector in order to get an improved signal-to-background ratio. The diffraction system was also equipped with an X-ray mirror that increased the intensity of the primary beam a factor of about 8. This particular system is tuned for a best dynamic range of $10^7$ and high resolution. The X-ray reflectivity and diffuse scattering experiments were carried out using a set-up consisting of the X-ray multilayer mirror (beam width 0.15 mm) and two slits 50 mm apart in front of the detector. Both slits were set to a width of 0.1 mm. The beam height was limited to 10 mm. In addition a secondary graphite monochromator was used in order to reduce the background. With this set up an instrumental angular resolution of 0.02$^\circ$ is achieved at a primary beam intensity of about 5.0 x $10^7$ cps. Diffuse scattering data were collected by scanning the incident angle at a fixed detector 20 position. Full pattern simulations based on dynamical scattering theory of diffraction [4] and total external reflection from multilayers [10] were used to analyze the data. The diffuse scattering was simulated by the Distorted Wave Born Approximation (DWBA), which takes into account the processes of refraction and multiple scattering [9].

**RESULTS AND DISCUSSION**

The diffraction pattern of a Si capped step graded two layers SiGe heterostructure is shown in Figure 1. This sample is a typical HBT device structure.

<table>
<thead>
<tr>
<th>Layer</th>
<th>Material</th>
<th>Thickness (nm) XRD</th>
<th>Thickness (nm) XRR</th>
<th>Roughness (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sub.</td>
<td>Si</td>
<td></td>
<td></td>
<td>0.4 ± 0.1</td>
</tr>
<tr>
<td>1</td>
<td>SiGe$_{0.161}$</td>
<td>55.1 ± 0.4</td>
<td>54.9 ± 0.3</td>
<td>0.4 ± 0.1</td>
</tr>
<tr>
<td>2</td>
<td>SiGe$_{0.051}$</td>
<td>28.9 ± 0.4</td>
<td>29.4 ± 0.3</td>
<td>0.37 ± 0.1</td>
</tr>
<tr>
<td>3</td>
<td>Si</td>
<td>38.1 ± 0.4</td>
<td>39.0 ± 0.3</td>
<td>0.2 ± 0.1</td>
</tr>
<tr>
<td>4</td>
<td>SiO$_x$</td>
<td>1.9 ± 0.6</td>
<td></td>
<td>0.2 ± 0.1</td>
</tr>
</tbody>
</table>

Table 1
Best-fit parameter values of XRD and XRR simulation for a HBT SiGe heterostructures.
The intense and narrow Si (004) substrate Bragg peak is accompanying by several weak asymmetrical peaks at the low angles. The pattern was simulated and fitted, assuming a model of two thin SiGe layers with different Ge content covered by a thin Si layer. The refinement involved in total 5 layer parameters (Table 1). An almost perfect match to the experimental curve was achieved (Figure 1) with this model indicating a well-defined structure with good homogeneity of the layers involved.

![Figure 1](image.png)  
*Figure 1* X-ray diffraction scan of a HBT structure (solid line) and full pattern simulation (dash-dotted line) around Si (004) reflection.

The analysis of the specular reflectivity curve (Figure 2) of this sample confirms the XRD findings.

![Figure 2](image.png)  
*Figure 2* Specular reflectivity curve and two diffuse scattering scans (solid lines) and the corresponding simulations (dotted lines) of a SiGe HBT structure.

A reasonable fit was achieved using three layers as in the diffraction model plus a thin oxide layer on top. It is the domain of XRR to reveal oxide or contaminant layers down to nanometer thickness. The initial density values of the SiGe layers were estimated from their lattice parameters as extracted from the diffraction pattern. The thickness values determined by both techniques (Table 1) show a reasonable agreement considering the different size of the illumination areas of...
the corresponding scans. The analysis of the reflectivity curve commonly also includes also a roughness parameter [10], but it is impossible to distinguish between interfacial roughness and density gradient since both have the same effect on the specular part. In order to extend the information about the in-plane structure non-specular or diffuse scattering scans are necessary. Two corresponding transverse scans, i.e. rocking curves at fixed detector positions, are depicted in Figure 2. The relatively sharp center peaks that are related to the specular reflected intensity dominate the scans. The symmetry and width of those is affected by the experimental set-up used and the macroscopic topology of the sample surface. Weak peaks appear at the angular positions where the incident or exit beam is close to the critical angle of total reflection. The presence of these so-called Yoneda wings is taken as indication for interfacial roughness [11]. To quantify the roughness parameters from the experimental diffuse scattering data the self-affine roughness model by Sinah et al [9] was employed. In this approach a rough surface is described by the root-mean squared vertical roughness and the lateral correlation length (i.e., the lateral distance at which the correlation has decayed to 1/e) and fractal dimension parameter H. The parameter H has a value between 0 and 1, and H close to 1 denotes a very smooth slowly oscillating surface while a value close to 0 describes a very jagged one. The calculated curve reasonably matches the measured data confirming the assumed fractal character of the surface (Figure 2). The shape of the diffuse scattering is mainly sensitive to the parameters of the two topmost layers. A corresponding correlation length of 85 ± 15nm and H value of 1.0 were obtained by the simulation using the roughness values extracted from the XRR.

Figure 3 Measured diffraction pattern (solid line) and best-fit simulations (dotted line) of SiGe layers with and without Carbon. The Ge content of both samples is about the same. Patterns are vertically shifted for clarity.

The second selected example describes the analysis of two SiGe:C layers both about 50 nm thick. In the SiGe base layers of HBT structures, carbon is incorporated with typical concentrations in the order of about 0.1%. XRD cannot measure the C concentration directly. Ge and C both affect the lattice parameter of a SiGe:C layer but in opposite directions while the XRD experiment accesses only the final strain value. Using calibration samples grown without C the Ge content can be extracted separately. The Ge value is then used to determine the C content in samples grown under the same growth conditions. SIMS studies on SiGe:C structures revealed that the Ge content is approximately constant dropping by about 0.3% per 0.1% incorporated C [12] at 16% Ge. In Figure 3 the diffraction pattern of two samples grown without and with C are compared. The
peak shift towards the substrate position is evident for the reduced layer strain by the C. Using a single layer model a good match of the full pattern simulations was achieved indicating a high structural perfection even for the structure containing Carbon. Based on the Ge content of 18.9% extracted from the carbon-free sample a C concentration of 0.24% was determined. Taking into account a possible slight drop of the Ge content in the carbon-containing sample the C concentration will yield 0.2%. Unexpectedly the reflectivity curves of these samples revealed a more complex behavior (Figure 3). The narrow spaced thickness fringes due to the layer thickness showed an additional modulation. From a Fourier transform analysis the corresponding thickness was estimated to be about 2.5 nm. A best fit of the reflectivity data could be achieved assuming a density gradient layer (density: 2.7 down to 1.6 g/cm³) of that thickness on top of the topmost SiGe epilayer. The results are summarized in Table 2.

<table>
<thead>
<tr>
<th>Layer</th>
<th>C: 0 %</th>
<th>C: 0.24%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>XRD</td>
<td>XRR</td>
</tr>
<tr>
<td>Si</td>
<td>d (nm)</td>
<td>0.35 ± 0.1</td>
</tr>
<tr>
<td>SiGe0.189</td>
<td>56.2 ± 04</td>
<td>56.6 ± 0.3</td>
</tr>
<tr>
<td></td>
<td>0.3 ± 0.1</td>
<td>0.15 ± 0.1</td>
</tr>
<tr>
<td>Graded layer</td>
<td>2.6 ± 0.3</td>
<td>2.0 ± 0.3</td>
</tr>
</tbody>
</table>

Table 2 Best fit values (thickness d and roughness σ) as derived from XRD and XRR simulations of nominally single SiGe layers with and without Carbon.

Based on this XRR model the simulations of the diffraction pattern were repeated but no significant improvement of the match of the single layer model to the experimental data was found. Such very thin gradient layer, even when perfectly crystalline, hardly affects the diffraction pattern but is easily detected by XRR. The diffuse scattering scans show an increased scattering intensity at the high angle Yoneda wing that cannot be described sufficiently by the diffuse scattering model that was used. The results of current experiments to address this observation will be published elsewhere.

Figure 4 Reflectivity and diffuse scattering curves (solid lines) and corresponding simulations (dotted lines) of a single layer SiGe structure without Carbon (upper curve) with 0.24% Carbon (lower curve). Curves are shifted vertically.
CONCLUSION

The combination of high-resolution X-ray diffraction, X-ray reflectivity and X-ray diffuse scattering has successfully proved to be a powerful tool to characterize structural features on a nanometer scale in epitaxial layer systems. The complementary information content of the used techniques is useful to study and solve growth problems not only of SiGe based structures but also all other types of epitaxial structures. Due to versatility of modern laboratory X-ray equipment, all types of scattering measurements can be easily performed on one single instrument without changing of the sample alignment. The availability of fast and reliable automatic refinement algorithms to simulate all experimental data significantly speeds up and simplifies the analysis process.

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