Strain distribution in Si capping layers on SiGe islands: influence of cap thickness and footprint in reciprocal space

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We present investigations on the strain properties of silicon capping layers on top of regular SiGe island arrays, in dependence on the Si-layer thickness. Such island arrays are used as stressors for the active channel in field-effect transistors where the desired tensile strain in the Si channel is a crucial parameter for the performance of the device. The thickness of the Si cap was varied from 0 to 30 nm. The results of high resolution x-ray diffraction experiments served as input to perform detailed strain calculations via finite element method models. Thus, detailed information on the Ge distribution within the buried islands and the strain interaction between the SiGe island and Si cap was obtained. It was found that the tensile strain within the Si capping layer strongly depends on its thickness, even if the Ge concentration of the buried dot remains unchanged, with tensile strains degrading if thicker Si layers are used.

(Some figures may appear in colour only in the online journal)
channel between source and drain [14, 15]. Several parameters are vital in the optimization of the strain properties of this channel: to maximize the tensile strain, a high Ge content within the stressor structure would be desired. In combination with a high Ge percentage a high aspect ratio of the stressor structure would even further increase the relaxation within the stressor itself and thereby the tensile strain within the Si layer grown on top.

In this paper, we focus on the influence of the Si-channel thickness itself, as this parameter can be refined concerning strain maximization and device processing. A sample series containing highly uniform, two-dimensional periodic SiGe island arrays capped with silicon layers of different thicknesses ranging from 5 to 30 nm was investigated by means of x-ray diffraction (XRD) experiments and finite element method (FEM) simulations. The combination of XRD experiments and FEM calculations was employed to get a realistic idea about the strain distribution within the SiGe islands and the Si layers on top. Parameters obtained from XRD measurements such as the average Ge content serve as input for strain calculations. The diffraction signal originating from the SiGe dots subsequently acts as the main fit parameter when XRD simulations based on the FEM models are compared to the experimental data. As the precise determination of Ge contents and strains directly from experimental data is hampered by the hydrostatic pressure that the Si cap applies to the buried dot [16], the FEM simulations provide crucial information on the degree of alloying within the SiGe islands and therefore their strain distribution—along with the thickness of the Si channel, these attributes significantly influence the strain properties of the entire structure and therefore the characteristics of any device built around such a capped island.

2. Experimental details

2.1. Sample growth and AFM studies

On 9 × 9 mm² pieces of silicon(001) substrate, square-shaped fields with a size of 400 × 400 µm² (width of unpatterned area between the fields 100 µm) were defined by electron-beam lithography and reactive ion etching, resulting in a regular 2D array of circular pits with a diameter of 180 nm and a depth of 80 nm. The periodicity of the pit-pattern was 800 nm. After a standard cleaning procedure [17], the substrates were introduced in a molecular beam epitaxy (MBE) chamber, where a 36 nm thick Si buffer was deposited at substrate temperatures which were increased from 450 to 550°C. Subsequently, five monolayers of Ge were deposited at 720°C with a growth rate of 0.03 Å s⁻¹. Dome-shaped SiGe island arrays with perfect uniformity were observed (see figure 1(a)) with one island per pit site. One sample remained uncapped; the rest were capped at 360°C no

will see in later sections of this paper this has consequences even for small substrate pieces.

The morphology of the dots was studied in detail by atomic force microscopy (AFM) operated in tapping mode. Those examinations revealed the presence of {1 0 5}, {1 1 3}, and {15 3 23} facets, which are characteristic for dome-shaped islands. No {0 0 1} top facet was found for uncapped islands. An AFM image of an uncapped SiGe island is depicted in figure 1(b) with the facets labeled. The aspect ratio (AR) of the islands (here calculated as island height divided by its base width) is about 0.2 with an average base width of 220 nm and a height of 44 nm. For the Si cap, the morphology was derived from AFM data as well. As soon as the capping layer thickness exceeds a certain limit, the junction of the four {1 0 5} facets starts to disappear, being replaced by a {0 0 1} top facet as seen in figure 2. This effect is already present in the case of the sample where the islands are capped with 5 nm Si. The AR of such a capped structure decreases slightly from the value of 0.2 for uncapped islands to 0.18 for islands capped with 30 nm Si, while no significant changes in the aspect ratio for smaller capping layer thicknesses were observed. From previous investigations it is well known that for a substrate temperature of 360°C no size and shape change of the buried SiGe islands occurs during Si capping [18–20]. Thus, the shape and size of both uncapped and capped samples as determined by AFM scans served as geometry input for the FEM simulations. Experiments involving selective chemical wet-etching and AFM scans performed on comparable samples revealed a core–shell-like Ge distribution as previously reported by Zhang et al [21]. Due to the rather high temperatures during Ge deposition of
720°C, complex intermixing processes take place in order to relieve compressive strain within the SiGe island. This results in a Ge-poor core situated at the center of the island base and a Ge-rich shell with a rather abrupt interface (see figure 1(c)).

2.2. X-ray measurements

XRD experiments were performed at the high resolution diffraction beamline P08 located at the Petra III synchrotron in Hamburg [22]. An energy of 8048 eV ($\lambda = 1.5406$ Å) was used; the incident beam was confined to a size of 200 $\times$ 200 µm$^2$ (in comparison the size of one patterned field was 400 $\times$ 400 µm$^2$). This eliminates contributions from islands on flat substrate areas surrounding the patterned fields sufficiently. A MYTHEN position sensitive detector (PSD) was employed to record the data. Reciprocal space maps (RSMs) including the symmetric (004) and the asymmetric (224) Si Bragg peak and the diffuse scattered intensities of the SiGe islands were recorded in coplanar geometry (see figure 3), by performing $\omega$–2$\theta$ 1:2 relative scans (see [23]). The integration time per step was 10 s, the stepwidth 0.005° for $\omega$ and 0.01° for 2$\theta$. The reciprocal space coordinates $Q_x$ and $Q_z$ (given in the units Å$^{-1}$) are obtained as the following:

$$Q_x = \frac{2\pi}{\lambda} [\cos(2\theta - \omega) - \cos(\omega)],$$

and

$$Q_z = \frac{2\pi}{\lambda} [\sin(\omega) + \sin(2\theta - \omega)].$$

Several remarkable features are visible in the recorded maps, especially regarding the (004) RSMs. First of all, a clear trend is visible comparing the measurements for different capping layer thicknesses: the width of the diffuse SiGe signals located at lower $Q_z$ values with respect to the Si bulk peak (marked
by an ellipse in the two uppermost panels of figure 3) narrows with increasing capping layer thickness. Accordingly, in the (224) RSMs it moves towards the crystal truncation rod (CTR). The so-called relaxation triangle in reciprocal space is defined by the bulk peak of the substrate lattice (in this case Si), then the position representing an alloy (SiGe) grown in a pseudomorphic way on that substrate (therefore situated somewhere on the CTR of the substrate due to the same in-plane lattice constants), and the completely relaxed state of that same alloy [24]. An island signal closer to the CTR therefore indicates a lower degree of relaxation for the buried SiGe island.

Strain values discussed in this paper are always given with respect to the corresponding bulk material in its relaxed state, i.e. by comparing the lattice constant of a SiGe alloy in the actual sample to the nominal value of the lattice constant of a relaxed SiGe alloy with the same composition:

$$\varepsilon = \frac{a_{\text{strained}} - a_{\text{relaxed}}}{a_{\text{relaxed}}},$$

(3)

where $a_{\text{strained}}$ is the lattice constant of a strained crystalline material as determined by an XRD experiment and $a_{\text{relaxed}}$ a relaxed bulk lattice constant of this material. Thus, strain values in the SiGe domains will always be displayed as negative (their lattice is compressed, meaning shorter lattice constant compared to the relaxed state), while the positive strain values represent the sections in the Si domains under tensile strain (elongated lattice constant with respect to the relaxed Si lattice). In-plane and out-of-plane strain values calculated from five datapoints evenly distributed over the length of the SiGe scattering signal show that the islands start out with comparable strain values at their base (associated with sections of the diffuse Ge signal close to the CTR) with roughly $-1.4\%$ compressive in-plane strain. Out-of-plane strains are approximately the same with reversed sign. Towards the island apex it becomes obvious that relaxation is suppressed more and more with increasing capping layer thickness. While up to a cap thickness of 5 nm nearly complete relaxation occurs at the island apex (meaning that both in-plane and out-of-plane strain values are approximately zero), islands capped with 30 nm Si still show compressive in-plane strain values of $-0.5\%$ at their apex.

Furthermore, in the symmetric (004) map can be observed. This is caused by the rather sharp interfaces separating sections with different Ge contents in the core–shell-like distribution discussed above (see figure 1(c) or Zhang et al [21]).

For the two samples with cap thicknesses of 20 and 30 nm, respectively, a distinct asymmetry of the SiGe signal in the symmetric (004) map can be observed. This can only be caused by a coherent tilt of the lattice planes within the buried island with respect to the bulk lattice, due to either the island geometry or Ge distribution being asymmetric. The actual cause for this effect will be discussed in section 4.2.

3. Finite element and x-ray simulations

While other experimental methods such as Raman spectroscopy have the problem of low penetration depth and therefore a strain sensitivity restricted to the upper 5 nm of the Si capping layer [25], XRD leaves us with rather the opposite of that problem. Due to the large penetration depth of the chosen coplanar geometry, a large volume of bulk material contributes to the scattered x-ray intensities recorded in an experiment. For the samples shown in this paper, the strained section of the Si cap above the SiGe islands does not provide enough scattering volume to produce a signal that can be distinguished from the diffuse scattering around the Si bulk peak. The SiGe signal, although stemming from a rather small amount of contributing volume, is well separated from the Si bulk peak. In the case of samples of simpler nature, for example homogeneous layers or uncapped samples, it would be straightforward to directly determine the Ge content and thereby the strain state directly from the XRD measurement. However, in our case we have to consider that the islands display a rather complex Ge distribution. Additionally, in the case of capped samples the Ge contents obtained directly from XRD experiments are incorrect due to the hydrostatic pressure applied by the Si cap [16]. Therefore, we use FEM models in combination with XRD simulations to determine the strain state of both the SiGe island and the Si cap.

To calculate the strain distributions, the commercial COMSOL Multiphysics program package [26] was used. The geometry for the FEM models was derived from AFM measurements; initial parameters for the Ge distribution were obtained from the XRD measurement of the uncapped sample and the etching profiles. The displacement fields obtained by FEM calculations also served as input for XRD simulations based on kinematical scattering theory [23]. These XRD simulations were then compared to the experimental data to refine the FEM calculations; the Ge distribution within the model island is altered until the SiGe signals in XRD simulations and measurements match sufficiently.

The shape and Ge distribution of the buried islands were determined based on the uncapped reference sample. The properties thus found were used for the buried islands in the models of the capped samples, meaning that for each model the same geometry and Ge distribution for the buried dot was used; only the thickness of the Si cap was varied (for an example of a model geometry, see figure 4(b)). The assumption that the shape of the buried islands does not change while capping at such low temperatures as used here is justified by previous XRD-based work [18] as well as TEM investigations on the 30 nm capped sample of this batch (see figure 8). In the model coordinate system the bottom of the downward facing pit pyramid below the island is situated at the origin, which makes it convenient to introduce analytical functions for the Ge distribution (see the inset of figure 4(a)).

A simplified Ge gradient resembling a core–shell-like distribution as seen in the etching profile (see figure 1(c)) was used; the Ge fraction within the island ranges from 33 to 43%. To mimic the core–shell gradient, an abrupt jump of 10% in the Ge content was introduced along a certain height parameter (in the center of the model island this jump is situated at a $z$-coordinate of roughly 40 nm, whereas the apex of the island is at 60 nm). This abrupt change in the Ge content also leads to a kink in the strain values, as can
be seen in figure 4(a). The resulting average Ge content was 37%. Several batches of FEM simulations were set up using such an island as base—one to obtain strain values within the Si channel for different cap thicknesses, one to investigate the asymmetric features in the (004) XRD reciprocal space maps features seen in the XRD maps and a further one with varied Ge contents to explore the maximum strain values achievable in the Si cap.

4. Results and discussion

4.1. Strain calculations for different capping layer thicknesses

The x-ray measurements shown in figure 3 indicate a trend towards less relaxation with increasing capping layer thickness. The thicker the Si cap, which is tensile strained and therefore wants to contract, the larger is the stress on the buried SiGe island. Due to the lattice mismatch between Si and Ge of 4.2%, the relaxation states of these two materials act counter-productively—a more relaxed SiGe island induces higher tensile strain in a Si capping layer, whereas the thicker the capping layer gets the more this layer itself is able to relax towards unstrained Si and thus compress the buried island by a higher amount. According to this, line plots of the in-plane strain based on the FEM models displayed in figure 4(a) show that, for islands with the same size, shape, average Ge content and Ge distribution, an increase of the capping layer thicknesses results in lower tensile strains in the Si cap while the overall compressive strain in the buried island increases. The SiGe signal in x-ray simulations based on these models (figure 4(b)) display the same behavior as the measurements which can be seen in figure 5.

The thin 5 nm Si layer on top of an island obviously displays the highest tensile strain with in-plane strain values around 1.3%, which would be desirable for the creation of strain-based devices. A thicker Si layer on the other hand, while being more relaxed with peak values around 0.8% for a 30 nm Si cap, is easier to maintain during device processing, as fabrication and etching steps can result in the loss of a few nanometers of the Si cap layer [15].

4.2. Origin of the asymmetric intensity distributions in (004) reciprocal space maps

In this section we discuss the cause for the asymmetric intensity distributions of the SiGe signal in the reciprocal space maps around the (004) Bragg peak mentioned earlier. An asymmetric intensity distribution around a symmetric Bragg peak as seen in the two lowermost sections of figure 3 can only be caused by a mutual tilt of the lattice planes of the probed material. In the case of the samples containing islands capped with 20 and 30 nm, respectively, only the island signal itself is affected (see figure 3), and the Si(004) substrate peak and the corresponding CTR are perfectly in line. This means that specifically the lattice planes within the SiGe islands are tilted with respect to the substrate in a coherent way throughout the entire illuminated sample area, otherwise such an effect would be averaged out due to the large number of probed islands (for an illuminated area of $200 \times 200 \mu m^2$ and a pattern period of 800 nm, roughly between $6 \times 10^4$ and $7 \times 10^4$ islands are included in the measurement). Reference measurements of the (004) Bragg peak in two perpendicular sample azimuths (both along $\langle 1 1 0 \rangle$ directions) performed at a laboratory rotating anode x-ray source confirmed that
Figure 5. In this figure XRD simulations based on the FEM models discussed in figure 4 are shown. In comparison to the actual measurement, these simulations show the same trend regarding the narrowing of the diffuse SiGe signal with increasing Si cap layer thickness (indicated by dashed lines). The SiGe signals shown here do not display the asymmetric behavior as observed in the experimental data (figure 3). The origin of this asymmetry along with corresponding simulations is discussed in section 4.2; see figures 6 and 7.

The asymmetric SiGe signal appears only along one [1 1 0] direction, while for the azimuth perpendicular to it the signal is symmetric.

There are several possible reasons for such a lattice plane tilt: one is an asymmetric Ge distribution, which has been observed for islands grown on flat surfaces [27, 28]. On patterned substrates this could be caused by a depletion effect [29], which occurs at the border between patterned and flat substrate areas: the predefined and favored nucleation sites in the pattern draw Ge from the surrounding flat substrate. Islands growing at the outskirts of a patterned field thus incorporate more Ge and develop in an irregular way compared to islands in the center of the pattern. In turn, a certain amount of the flat area surrounding the pattern is depleted of Ge; no islands nucleate there. However, in the case of the uncapped sample and the ones with 5 and 10 nm Si cap, no asymmetry of the SiGe signals is visible in the (004) RSMs (see figure 3); therefore, the tilted signal cannot be caused by the SiGe islands themselves. This rules out the depletion effect or an asymmetric Ge distribution due to some other cause.

This leads to the conclusion that the Si cap itself is the cause of the asymmetric intensity distribution we see in our XRD measurements. AFM scans of capped samples did not show shape irregularities of the Si cap in a preferential direction; its surface looks perfectly symmetric, as shown in figures 2(c) and (d). However, as no sample rotation was used during the whole growth process, the complete cap could be shifted with respect to the buried island due to the geometric setup in the MBE chamber. The beams of both Si and Ge adatoms do not impinge on the sample vertically; in the case of the Si source, the inclination angle with respect to the sample surface normal is approximately 20°.

This had no detectable effect on the Ge distribution of the islands due to the higher mobility of Ge adatoms to begin with and the higher growth temperature of 720 °C during Ge deposition. The diffusion rate of Si adatoms is much smaller; additionally, a lower substrate temperature of 360 °C was used during the capping process. This lead to a slightly higher Si coverage on the island slopes facing the Si source, while the overall cap shape still looks symmetric. This inhomogeneous coverage, which appears to be more or less uniform for the entire island array, leads to a mutual tilt of the lattice planes within the SiGe islands, resulting in a signal that is tilted with respect to the (0 0 1) axis in reciprocal space. Corresponding FEM models were set up for the samples with 20 and 30 nm Si caps with an island displaying a perfectly symmetric Ge distribution and a Si cap with a lateral shift with respect to the buried island (see figures 6 and 7, respectively). Shifts ranging from 5 to 15 nm were tried (in 5 nm steps), where a shift of 10 nm leads to a significant tilt of the SiGe signal in the x-ray simulations, comparable to the effect seen in the experimental data. The direction of the tilt in the RSM depends on the sample orientation with respect to the incident x-ray beam.

The asymmetry of the islands’ x-ray signal is more pronounced in the sample with the thinner cap of 20 nm. This is clearly visible in the XRD measurements if the RSMs for the 20 and 30 nm capped samples are compared. In the case of the 20 nm sample the shift of the SiGe signal is strong enough to place it almost entirely to one side of the truncation rod, while for the 30 nm sample some diffuse intensity can be observed on the opposite side of the truncation rod.
Figure 6. FEM simulation results for SiGe islands capped with 20 nm Si. (a)–(d) Maps for the in-plane strain, the displacement field along the $x$-direction, the out-of-plane strain and the $z$-displacement, respectively. The 2D plots are shown along the [1 1 0]/[0 0 1] direction; the coordinates are given in nanometers. The displacement fields, which are used as input for the XRD simulation, clearly show an asymmetry effect. The values for the geometric grid are given in nanometers. The fit for simulated RSMs (contour) based on this model with the experimental data (colorplot) is shown in (e) for both the (004) and the (224) Bragg peak.

Figure 7. In-plane strain (a), $x$-displacement (b), out-of-plane strain (c) and $z$-displacement (d) of the FEM model with a 30 nm cap. The asymmetric displacement field is a little less pronounced than for the sample with 20 nm due to the thicker cap in this case. This is also visible from XRD simulations (contour) and measurements (colorplot) shown in (e); the shift of the diffuse SiGe signal is slightly weaker than for the 20 nm sample.

As the Si cap gets thicker, it applies more and more hydrostatic pressure on the buried island. It is very likely that at a certain cap thickness the pressure applied by the cap becomes homogeneous again; i.e., a minor difference in the Si coverage on the respective island slopes is of no further significance if the cap is thick enough. The assumption concerning the shifted cap was confirmed by TEM investigations. The images shown in figure 8 reveal that both island and cap are of symmetrical shape, but their centers are indeed shifted by approximately 10 nm with respect to each other. This results in different Si coverages on the respective slopes of the island, which of course has a pronounced influence on the island's overall strain field. Aside from this, it can also be seen that the island’s $\{1 0 5\}$ top facets are still perfectly shaped after Si capping; no flattening of the island apex is observed. Of 23 investigated islands on the lamella, only the one closest to the edge of the TEM lamella contained stacking faults, which is probably related to the sample preparation process. The investigated volumes in the other islands as well as the caps were found to be defect free (see figure 8(c)).

4.3. Comparison of strain values

In figure 9 two plots for the in-plane strain versus the capping layer thickness are shown, visualizing the trend described above. The upper panel displays the strains for the cap: both maximum strain tensile strains are shown as well as an average value obtained by integration over the volume of
Figure 8. Bright-field STEM image of an island with 30 nm Si capping layer (a). Both island and cap appear to be symmetric but shifted by about 10 nm with respect to each other. This shift results in different Si coverages on the slopes of the island, affecting the strain field in an asymmetric way and thereby causing the asymmetrical SiGe(004) signal in the measured RSM. (b) A high angle annular dark-field STEM image to visualize the material contrast between Si (dark) and Ge (light). (c) The same image with a defect-sensitive contrast: a minor amount of impurities along the interface between the original Si substrate and the buffer layer is visible; the buried island as well as the Si cap are defect free.

Figure 9. In-plane strains versus the capping layer thickness for all simulations performed for this sample series. The upper section displays the maximum tensile strain (red line) achieved within the Si cap, whereas the green line represents average tensile strain values. The blue circles are corresponding datapoints for the models with a shifted cap. In the lower panel the compressive average in-plane strain values for the buried island with respect to the capping layer thickness are shown.

Figure 10. Simulation results for FEM models containing dome-shaped SiGe islands capped with 30 nm Si with varied Ge content. As template, the core–shell distribution and geometry discussed earlier (220 nm island diameter, 44 nm height) were used with an offset in the Ge gradients to obtain islands with different average Ge contents. The average Ge fractions were varied from 20 to 94%; in the latter case the island shell already consists of pure Ge. Here, a maximum tensile strain of 2% could be achieved within a 30 nm thick Si cap.

to alter the overall Ge content. In the calculations, the Ge percentage was increased until the Ge-rich shell within the island consisted of pure Ge (with the used gradient, this corresponds to an average Ge content of 94%). For all the simulations a Si cap thickness of 30 nm was used. Similar to the simulations discussed earlier, these models are based on a purely elastic approach as well, not taking into account plastic relaxation caused by dislocations.

The results of these simulations are shown in figure 10. For the specific Ge distribution used here, maximum tensile strain values of 2% were found in the cap if the island shell consists of pure Ge. Of course, this changes significantly if the Ge gradient or the island shape is altered. The Ge contents for the samples investigated in this paper are still relatively low, with an average value of 37%. The Ge fraction could be increased by growing the islands at lower temperatures and/or closely stacking two Ge islands on top of each other.
as described by Zhang et al [30]. In this specific case, Ge contents up to 50% were achieved, which would result in a tensile strain of 1.57% in a 30 nm thick Si cap. Of course, lowering the growth temperature has to be treated with care, as dislocations are introduced more easily. This means that SiGe islands as stressors are able to induce high tensile strains in defect-free Si layers, still leaving room for improvement on behalf of the stressor structure itself and the possibility to further enhance the strains by combining this technique with stressed nitride layers for example.

5. Summary and conclusion

To summarize this paper, we presented XRD measurements, strain calculations and XRD simulations for a series of SiGe islands grown on 2D-periodic prepatterned Si(001) substrates and capped with different amounts of Si. Strain calculations show that maximum tensile strain values within the Si capping layer decrease from 1.3% to 0.8% for cap thicknesses ranging from 5 to 30 nm. Furthermore, the XRD measurements revealed that due to the absence of sample rotation during MBE growth the Si cap is displaced with respect to the buried island. This shift was identified as the source of asymmetric SiGe signals that appeared in the RSMs around the symmetric Si(004) Bragg peak. While in AFM scans providing information on the surface of the sample the shape of both uncapped and capped islands looked perfectly symmetric, XRD and TEM investigations revealed their asymmetry. We prove that with XRD we are able to detect certain irregularities in the investigated nanostructures as well as to explain and reproduce them with model simulations. XRD proves to be an excellent tool to detect alterations of the strain state within SiGe islands if different amounts of Si cap layer are deposited, or if the thickness of this capping layer is inhomogeneous. We were able to reconstruct the experimental XRD data by performing x-ray simulations based on FEM models with realistic properties and thus obtain information on the strain fields of both SiGe islands and Si cap layers. Calculations based on the geometries determined during this work featuring increased Ge contents showed that 2% maximum tensile strain is achievable in 30 nm thick Si caps grown on dome-shaped islands if the island shell consists of pure Ge. This renders SiGe dots as stressors for Si channels as used in MOSFET technology to be a valuable technique with still some potential to exploit.

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