GERMANIUM HUT NANOSTRESSORS ON FREE-STANDING ULTRATHIN SOI

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Abstract. Heteroepitaxial growth of SiGe on thin Si membranes leads to the sharing of the epitaxial strain between the Si template layer and the deposited thin film. At high Ge concentrations, at which Ge forms dislocation-free hut nanostructures, there can be significant bending underneath self-assembled Ge huts. We have fabricated undercut mesas to approximate a freestanding Si membrane and produced Ge hut structures using molecular beam epitaxy. Using synchrotron x-ray microdiffraction to probe the strain and bending of the template layer directly, we compare strain sharing in conventional blanket film structures with the strain induced by Ge hut structures.

INTRODUCTION

A new generation of Si electronic devices depends on the control of large lattice strains (up to a few percent) in devices at length scales below 100 nm.[1] A conventional approach has been to fabricate compositionally graded SiGe alloy thin films that apply strain to a subsequent epitaxially grown Si layer. Recently, similar structures fabricated on thin SOI substrates have used a SiGe alloy thin film to induce percent-level strains into the template layer after mechanically freeing it from the substrate.[2] The unique mechanical situation associated with a thin, mechanically compliant substrate changes the conventional description of the role of strain in the growth of thin films. The concept of a critical thickness for the propagation of misfit dislocations, for example, is drastically altered on thin SOI membranes. As a strategy for creating novel mechanical nanostructures, decoupling strained multilayer thin films from a substrate can result in a rolling of the films into tubes with diameters less than 100 nm [3] – and will be the basis, in the future, for more elaborate structures.

Recent advances in producing thin, mechanically isolated crystalline layers, particularly in SOI structures, have lead to a wider range of new phenomena. The thickness of the Si template layer in a SOI structure can be reduced to the point where partitioning of the total epitaxial mismatch strain between the substrate and the film is important. Decreasing the template layer thickness, however, also blurs the distinction between quantum-dot-scale stressors, which are typically thought to produce purely local distortions,[4] and blanket films that produce large-scale wafer curvature.[5] The length scale at which films can be considered large in a lateral direction is set by the thickness of the template layer. For ultrathin SOI with thicknesses of tens of nanometers, this length

scale is smaller than even fully coherent self-organized quantum dots. The mechanical response of conventional substrates, with thicknesses of many microns, has already been shown to be a powerful tool for understanding stress evolution in SiGe thin film growth at macroscopic lateral scales.[6] We have developed microfabricated structures with free-standing Si layers to probe both conventional strain sharing using blanket films and also to understand the strain imparted on a Si layer by a Ge quantum dot.

FABRICATION OF SILICON MEMBRANES

For studies of strain sharing in the conventional blanket film geometry, Si membrane windows with lateral dimensions 0.5 mm × 0.5 mm and thickness 140 nm were prepared on SOI following conventional lithographic processing. Commercially available SOI substrates with 200 nm Si template layers and 200 nm buried oxide layers were used as the starting substrate. A dry thermal oxidation was performed to protect the surface of the Si template layer during processing. PECVD Si_3N_4 was deposited on all sides of the oxidized wafer to act as an etch stop. Photolithography was used to pattern large squares on the back side of the wafer, the nitride and oxide were removed from the squares using dry etching, and the substrate was etched with KOH until the buried oxide was reached from the backside. The nitride resist was then removed by wet etching with phosphoric acid and the oxide was removed with HF. The remaining substrate consisted of an array of square 150 nm-thick crystalline-Si films in "windows" confined at all edges on the surface of the wafer.

Free-standing regions to be stressed by Ge huts were formed as undercut Si mesas where the edges were 30 nm thick free-standing Si, but the center was still rigidly attached to a thick substrate. Using bonded SOI with a 110 nm Si-template layer and a 1.3 micron buried oxide layer obtained from SIGen, a sacrificial dry thermal oxide was grown at 1050 ºC and removed with an HF dip, thinning the Si template layer to 30 nm. The Si template layer was patterned into 5 μm square mesas using optical lithography and dry etching with a SF_6 plasma. The oxide under the edges of the mesas was etched using HF vapor to create a freestanding ~250 nm wide region. The vapor etch was conducted with the HF at room temperature, while the substrate temperature was elevated slightly using an incandescent lamp.[7]

X-ray microdiffraction studies of strain in these micromachined structures were performed at the dedicated x-ray microfocusing facility at station 2ID-D of the Advanced Photon Source at Argonne National Laboratory.[8] In contrast with other tools often used to study strain in micron-scale devices, such as optical interferometry or Raman scattering, x-ray diffraction is an inherently structural technique that couples directly to the distortion of the lattice. We collected diffraction patterns for the SOI template layer's Si (004) reflections using a charge-coupled-device (CCD) camera. The CCD images were converted into conventional reciprocal-space maps, from which the strain and bending in the template layer could be measured. The x-ray measurements were performed using a photon energy of 11.2 keV and a beam focused to a spot size of approximately 200 nm at the sample.

STRAIN SHARING IN SILICON MEMBRANES

The x-ray reciprocal-space maps in Figure 1 illustrate strain sharing in a membrane created by depositing 280 nm of $Si_{0.95}Ge_{0.05}$ on both sides of a 150 nm thick Si-film window at 625 ºC using chemical vapor deposition (CVD). At this temperature, the growth of the SiGe thin film results in a fully coherent heterostructure both within and outside the window regions. The Si (004) x-ray reflection from the handle wafer and the attached template layer appears near $2\theta=48.1°$ and corresponds to undistorted Si lattice planes. The SiGe (004) reflection at slightly lower angle indicates that the SiGe film has grown epitaxially without strain relaxation due to defect formation. In the unsupported-membrane region, both the Si and SiGe reflections are shifted to higher angle as the strain due to the SiGe layers is shared with the template layer. The strain sharing can be predicted quantitatively using either a force balance [2] or by minimizing the coherency energy.[9] Based on the composition of the SiGe thin film, the expected biaxial strain in the template was 0.12%, which is in excellent agreement with the 0.14% measured using the data in Figure 1.

On a separate window, a 300 nm $Si_{0.90}Ge_{0.10}$ film was grown on only one side using molecular beam epitaxy at a sample temperature of 625 ºC. The biaxial strain deduced from the x-ray microdiffraction measurements, shown in Figure 2, was 0.20%, in agreement with strain sharing predictions. Despite its lower thickness, the higher composition of the film grown by MBE strains the template layer more efficiently than the films grown by CVD because the strain induced by a relatively thick film into a thin layer increases very slowly with film thickness.

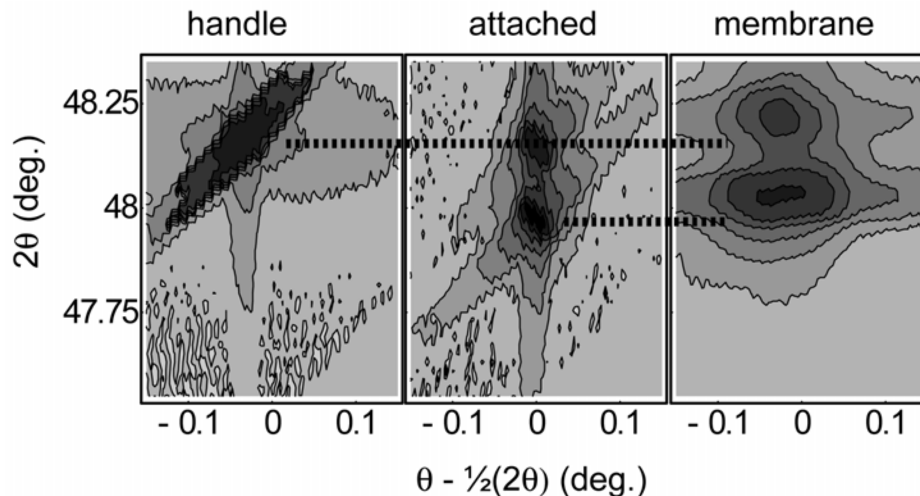

Figure 1. Reciprocal-space maps of the Si (004) x-ray reflections of the handle wafer, an attached SOI template layer, and an unsupported membrane. Dark areas are regions of higher x-ray intensity.

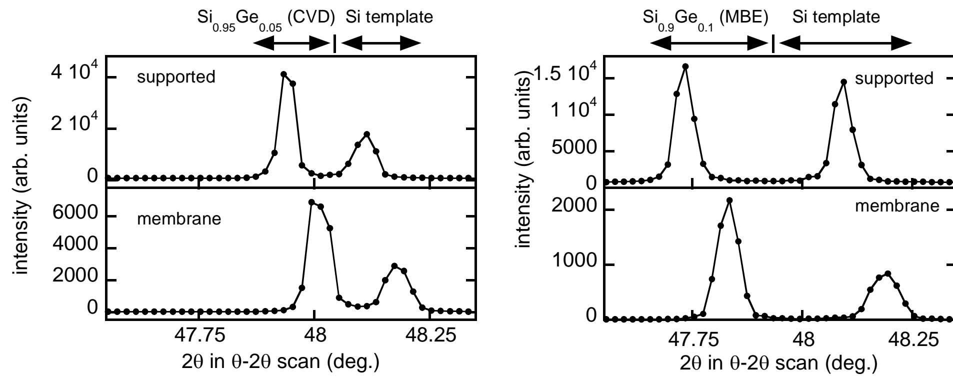

Figure 2. Theta-two theta scans computed from x-ray microdiffraction reciprocal space maps of $Si_{1-x}Ge_x$ films grown by CVD (x=0.05, left panel) and MBE (x=0.1, right panel).

GERMANIUM HUT NANOSTRESSORS

Ge huts were grown on the microfabricated undercut Si mesas using MBE. A Ge film approximately 4 monolayers (ML) thick was deposited at a substrate temperature of 550 °C at a rate of 0.5 ML min^{-1} by panning a shutter across the sample during the first 30 seconds of growth. The time during the growth at which the onset of hut formation occurs was previously determined by using RHEED to monitor the surface of a Si substrate. As it can be difficult to stop the growth following the transition at precisely the desired hut density, a range of growth times around the 2D-to-3D transition ensured that some part of the sample would have optimal hut structures. A schematic diagram of the distortion of the SOI template layer and a scanning electron micrograph illustrating the distribution of Ge huts near the edge of a Si mesa are shown in Figure 3.

The distortion of the (free-standing) Si template layer by the Ge huts broadens the x-ray diffraction pattern of the free-standing region. The diffraction pattern of an identically fabricated free-standing region which was shadowed during the Ge deposition to serve as a control is sharper than the diffraction pattern of the region with Ge. The intensity of the diffracted beam as a function of the angle perpendicular to the scattering plane has a width of 0.5° (Figure 4). The full width at half maximum (FWHM) in the same direction of the reflection from the region without Ge was 0.08°, slightly broader than the resolution set by x-ray optics used to create the focused x-ray beam, but much narrower than the distorted region.

The x-ray diffraction results can be interpreted to find the curvature of the template layer in the region beneath the Ge hut structures. A useful approximation is to assume that each of the few huts illuminated by the submicron x-ray beam is identical and that the bending of the template layer occurs only beneath the 50 nm lateral size of the hut. A distortion of 0.5° over that distance corresponds to a radius of curvature of approximately 6 μm, which can be compared to mechanical predictions of the response of the substrate to a localized stress. Continuum finite element calculations and atomistic molecular dynamics models provide the strain distribution in the template layer near the island, but do not provide an overall estimate of the total stress on the template layer.[4,10,11] In

the simplest sense, the curvature induced in a thick substrate by an infinitesimally thick blanket film is given by the Stoney equation, $\kappa_{st} = 6m\varepsilon_m h_f / h_s^2$. Here h_f and h_s are the thickness of the film and substrate and ε_m=0.042 is the epitaxial mismatch between Si and Ge. The ratio of biaxial elastic constants is m=1.3 for Ge on Si.[12] A naïve use of the Stoney equation with the thickness of the template layer (30 nm) and average height of the huts (1.6 nm) gives a radius of curvature of 1.7 μm. The correction for finite thickness substrates derived by Freund *et al*. [5] increases the radius of curvature by approximately a factor of $(1+h_f/h_s)^3$, about 15%. A further correction can account for the shape and finite size of the hut cluster, but results in only a few percent change in the curvature.[13,14] The remaining difference between the slightly larger experimentally observed radius of curvature and the predicted values may be due to oxidation of the uncapped Ge huts following their removal from the MBE chamber before the x-ray measurements.

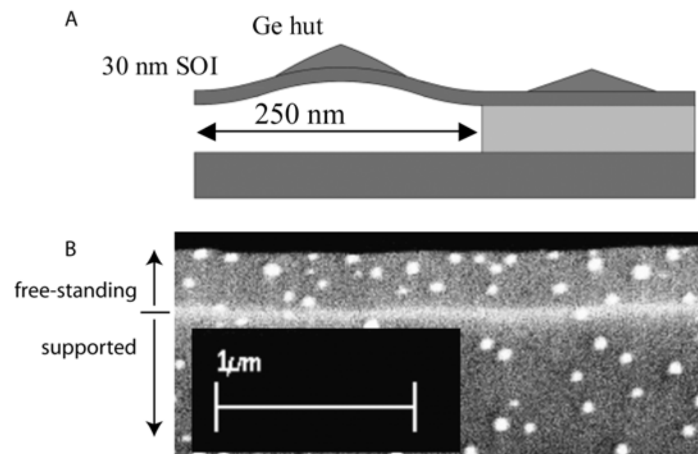

Figure 3. Sub-100 nm Ge hut nanostructures on a free-standing 30 nm-thick Si layer, (A) schematic cross section and (B) top view scanning electron microscope image. The SOI template layer is mechanically supported along the bottom edge of the image.

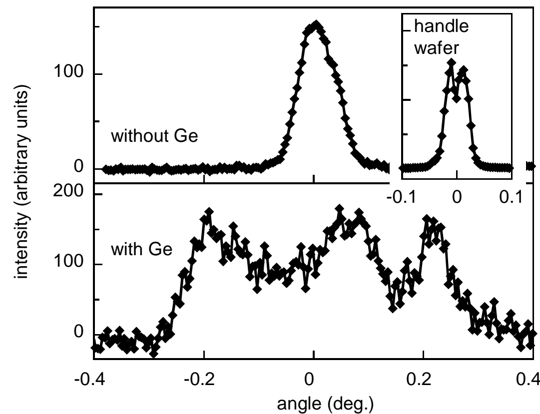

Figure 4. The Si (004) x-ray reflection from a free-standing SOI template layer is broadened by Ge huts. The width of the diffraction peak from the region with huts (lower panel) is several times those from regions without Ge (upper panel) and from the handle wafer (inset).

CONCLUSION

The Si template layer in SOI structures can be thin enough that the conventional mechanical roles of substrate and thin film in epitaxial growth do not apply. For continuous 2D films, the relative thicknesses of the substrate and film can even be reversed, as in Figure 1, making a quantitative understanding of strain sharing phenomena imperative. For thinner template layers, quantum dots effectively become large enough to be nearly approximated as continuous films in curving the template layer. In both cases, the distortion can be seen quantitatively in the broadening and shifting of x-ray reflections from the SOI layer. The large localized strains induced by hut nanostressors in particular have the potential to be an important addition to the emerging set of experimental tools exploiting the electronic effects of strain in Si.

ACKNOWLEDGEMENTS

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