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DIRECT SYNCHROTRON X-RAY MICRODIFFRACTION MEASUREMENTS OF STRAIN AND BENDING IN MICROMACHINED SILICON DEVICES

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ABSTRACT

The manipulation of strain in micromachined silicon structures is an important aspect of the design of emerging mechanical and electronic devices. Strain also has a fundamental role in the formation of devices through its effects on surface processes in epitaxial growth including diffusion and can be an important tool for studying these processes. Microfabricated silicon structures offer the opportunity to control the strain at length scales of less than one micron to several hundred microns. Synchrotron x-ray microdiffraction allows simultaneous independent measurements of the strain and bending in these structures. Microdiffraction measurements show that bending is the dominant source of strain in a prototypical microfabricated Si bridge loaded at its ends by silicon nitride thin films. The total strain difference between the top and bottom of the bent bridge exceeds 0.1% in our prototype structures and can potentially be increased in optimized devices.

INTRODUCTION

The distortion of a crystalline material can have a dominant role in the behavior small mechanical and electronic systems. A new generation of high-speed transistor devices depends on the control of large lattice strains (up to a few percent) in devices at the 100 nm scale.[1,2] 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 as small as 100 nm.[3,4] In condensed matter physics, a simple distortion of the lattice can be sufficient to change the phase of a complex oxide phase between paramagnetic and ferromagnetic [5], or to induce ferroelectricity in a nominally cubic crystalline thin film [6] A similar distortion of the lattice can be driven by an applied electric field in piezoelectric solids. X-ray microdiffraction, an emerging quantitative technique, is an ideal probe of strain and bending in micron-scale structures. An important result of the ability to measure strain quantitatively and precisely in the small structures is the possibility to engineer devices with built-in or even dynamically adjustable strain. 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.

SYNCHROTRON X-RAY MICRODIFFRACTION

Our x-ray microdiffraction studies of strain in silicon micromachined devices were performed at the dedicated x-ray microfocusing facility at station 2ID-D of the Advanced Photon Source at Argonne National Laboratory.[7] The Advanced Photon Source is a third generation synchrotron radiation facility, optimized to produce a spatially small source of x-ray radiation with a small angular divergence. At station 2ID-D, a monochromatic x-ray beam is produced by a Si (111) double crystal monochromator and focused to a spot with a Fresnel phase zone plate lens.[8] An x-ray beam can be focused in this way to a spot approximately 100 nm in diameter. One consequence of focusing the x-ray beam is that the numerical aperture of the lens introduces a divergence of approximately 0.05° into the focused beam. The rocking curves of single crystals are effectively broadened to this value.

For our diffraction experiments a sample was mounted at the center of a six-circle kappa-geometry diffractometer and the center of the diffractometer was aligned to the focal point of the

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beam (Figure 1). Specific regions of the sample were studied in diffraction measurements by moving the sample to bring the areas of interest into the beam. The measurements presented here were made using a zone plate with a nominal focal length of 10 cm to focus 11.2 keV x-rays. A charge-coupled device x-ray detector recorded the angular position and intensity of the diffracted beam.

Yang *et al.* have performed similar measurements on milimeter-scale bent silicon single crystals using a polychromatic x-ray beam focused using mirror optics.[9] In comparison with our monochromatic technique, however, using polychromatic x-rays can introduce additional complications in the interpretation of the results. X-ray microdiffraction with larger focused spot sizes has also been used to evaluate crystal distortion precisely in the investigation of cm-scale mechanical structures.[10]

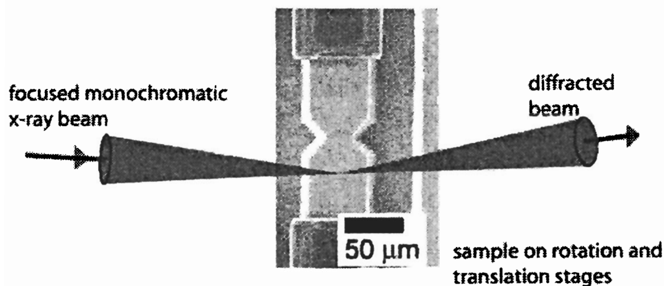

Figure 1 X-ray microdiffraction using Fresnel zone plate focusing optics.

X-ray microdiffraction is a broadly useful tool that takes advantage of the large variety of ways that x-rays can interact with solid materials. In addition to these measurements of strain in MEMS-inspired structures, x-ray microdiffraction has recently been extended in our experiments to study magnetic domains in antiferromagnetic metallic chromium and to probe the switching of stored polarization in ferroelectric thin film capacitor devices [11,12].

MICROFABRICATED STRAINED SILICON STRUCTURES

The silicon structures for this study were fabricated from silicon-on-insulator (SOI) wafers using silicon micromachining techniques. Briefly, top, device, layer of the SOI was patterned using photolithography and etched with chemical or reactive ion etching. The resulting structures were freed from the substrate using hydrofluoric acid to dissolve the intermediate layer of SiO_2. Strain was induced at specific locations in these structures using a patterned silicon nitride film grown by chemical vapor deposition. Varying the parameters of the silicon nitride deposition can control the degree of strain. The devices of Figures 1 and 2 were formed in SOI wafers with a 9 μm-thick device layer and were stressed by silicon nitride films approximately 1 μm thick.

Simple microfabricated cantilevers (Figure 2) were produced with silicon nitride stressor layers, which are visible as a raised area at the connected end of the cantilevers. These cantilevers bend upwards from the substrate, in a direction that curves the silicon *towards* the silicon nitride layer. The strain in the nitride layer is thus tensile following deposition. The silicon nitride films deform the substrate on which they are deposited and, as in this case, can lead to large deformations in thin substrates.[13,14]

We also fabricated bridge structures (Figure 1) in which both ends of a bar were stressed by silicon nitride layers. The central 150 μm of the bridges was exposed and bent upwards. A notch at the center of this span served to concentrate the bending. The device in Figure 1 in particular was designed to have well defined strains at the exposed top surface of the bridge. These strained silicon surfaces are ideal devices for studies of the effect of strain on the fundamental processes of epitaxial growth.[15]

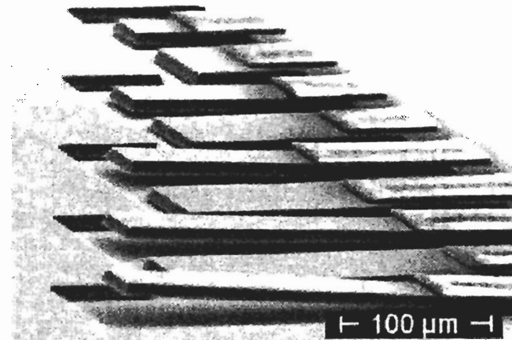

Figure 2 Microfabricated cantilevers raised by a strained silicon nitride layer.

This approach can be extended to small length scales using thinner SOI device layers and spatially smaller sources of strain. Recently, similar structures fabricated on far thinner SOI substrates have used a silicon-germanium alloy thin film to reach strains at the level of percent.[1,16] An extreme limit of this approach is strain developed by self-assembled strained Ge hut structures. The huts have typical dimensions of tens of nanometers. Due to the lattice mismatch between Si and Ge, the huts can lead to large strains in the silicon layer.[17] Figure 3 shows a plan-view scanning electron micrograph of Ge huts at the edge of a 30 nm-thick silicon layer supported along the bottom half of the image. Our x-ray microdiffraction measurements using a similar approach to the one described here, to be published elsewhere, indicate that the Ge huts produce large strains in the underlying silicon.

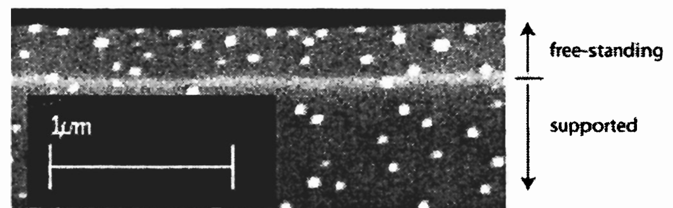

Figure 3 Sub-100 nm germanium hut nanostructures on a free-standing 30 nm-thick Si layer.

DIRECT X-RAY MEASUREMENTS OF STRAIN AND BENDING IN A SILICON BRIDGE

Because the silicon devices of Figures 1 and 2 are formed from single crystals of silicon, distortion in the devices leads to bending and strain in the silicon lattice. The silicon bridges were formed with a crystallographic [110] direction along the length of the bridge and a [001] direction normal to the surface. By measuring the small differences in this orientation in different places of the device the bending of the silicon layer

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can be deduced. In principle this can be accomplished with any set of planes with orientations that are changed by bending.

We used the silicon [004] reflection to find the tilt of the planes of the silicon across the bridge. The tilt of planes can be deduced by aligning the sample as shown in Figure 1 and scanning the beam along the length of the bridge. In regions where the bridge is tipped out of the plane of the surface the beam will be deflected in the direction along the length of the bridge and the tilt of the planes can be deduced. In Figure 1, the this tilt increases from zero as the sample is bend upwards by the silicon nitride layer (which begins at approximately a position -300μm from the notch) and reaches a maximum magnitude at the edge of the nitride. The free region of the bridge has opposite curvature and reaches the opposite tilt to match the bending induced by the other nitride layer. The curvature of the bridge, which is found from the rate of change of the tilt with position, is higher in the region of the notch at the midpoint of the span of the bridge.

The sample can be aligned in a slightly different geometry to make the same measurement using the silicon (022) reflection. This reflection must be observed in an arrangement where the diffracted beam is transmitted through the sample because the (022) direction lies either in the plane of the surface of the sample or within a small misorientation of it. The tilt of the maximum in the silicon (022) rocking curve can be used to find the tilt of the planes and gives results consistent with Figure 3.

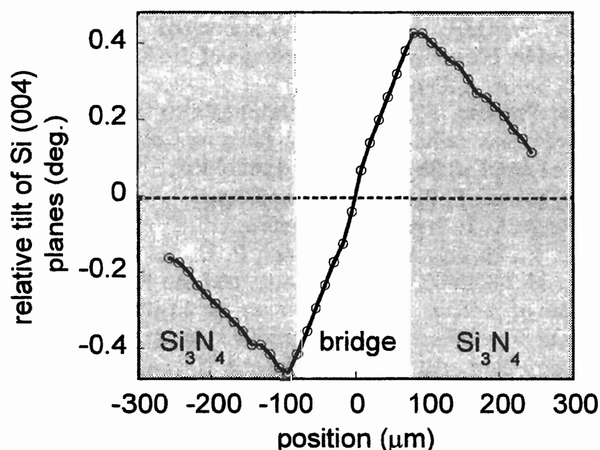

Figure 4 Bending measurements for microfabricated bridge.

The surface strain that the bridge structures were designed to produce can be deduced from the bending of the planes by a simple geometrical argument. A fixed number of planes of silicon atoms must span either a greater distance (on top of the bridge) or smaller distance (on the bottom) due to the curvature. This argument, however, assumes that there is no relaxation of the strain by crystal defects; i.e. we must assume that the bending is purely elastic. Similar measurements have been used to measure wafer curvature induced by thin films on length scales of one millimeter or more using laboratory x-ray diffraction techniques.[18]

A second approach to the diffraction experiment can complement the bending measurements by giving a direct measurement of the strain in the lattice. The angle to which the x-ray beam is diffracted depends on the wavelength of the x-ray and the spacing of the diffracting planes. The angle between the diffracted and incident x-ray beams, conventionally called 2θ, thus varies slightly in regions where crystals are strained. Conveniently in our case, the top and bottom surfaces of the bridge diffracted *simultaneously*. The diffracted beam for the Si (220) reflection became broader in 2θ in regions where the lattice was strained. By using the diffracted beam 2θ-angle to measure the strain directly this method does not need to make the approximation that the strain is elastic. The difference in the lattice constants on the top and bottom of the bridge can be deduced from the magnitude of the broadening. The two measurements of the strain, based on curvature and the broadening of the (022) reflections are in excellent agreement. The strain at the surface of the bridge is more than 0.1%. This is much less than the ~4% that can in principle be produced by Ge epitaxial layers but it is sufficient to have a large effect of surface phenomena.[19]

BENDING INDUCED BY NITRIDE STRESSOR LAYERS

The strain in our structures is developed in the thin silicon nitride layer deposited on top of the SOI device layer. The degree of strain in these types of films is often calibrated by measuring the curvature the strained film induces in the single crystal substrate. In doing this, the substrate functions simply as a well-characterized elastic solid. Typically optical curvature measurements are used. When the thickness of the film is known, the curvature can be used to find the average stress within the thin film using the Stoney equation.

These optical measurements and related approaches using laboratory x-ray techniques [18] cannot be applied after the stressor layers are patterned into devices. Microdiffraction allows the stress within the thin stressor layers to be estimated even after processing into very thin devices. We also used the curvature of the elastic solid beneath the nitride layer – in this case the stressed region of the bridge – to deduce the stress in the nitride layer. We measured the curvature in the direction across the bridge, where we expect the constraint due to the presence of the bridge to be minimized. The bending of the bridge in this direction shifted the center of the (004) reflection rocking curve by 0.1° in the central 25 μm of the span (Figure 5). Using the tabulated elastic constants for silicon, this gives a stress in the nitride layer of 190 MPa. This is approximately 15% less than the value measured for blanket films fabricated in the same way, perhaps due to etching of the nitride film during the patterning steps.

Two important experimental subtleties occur in the data shown in Figure 5. At the edges of the bridge, at the large and small values of the position, the rocking curve angle does not depend on the position of the beam. This effect occurs because the silicon nitride film covers only the central ~35 μm of the silicon bridge and is not in contact with the bridge at its edges. The substrates is curved only in places where the silicon nitride is attached. In addition, the bridge appears to be wider than 50 μm based on the data from Figure 5 because in the geometry shown in Figure 1 the x-ray beam strikes the sample at an angle equal to the Si (004) Bragg angle, approximately 20°. Along the direction across the beam, the beam footprint on the sample is longer than the spot size and the effective resolution of the measurement is lowered. Lower resolution in scans in this direction because the beam penetrates the full thickness of the sample.

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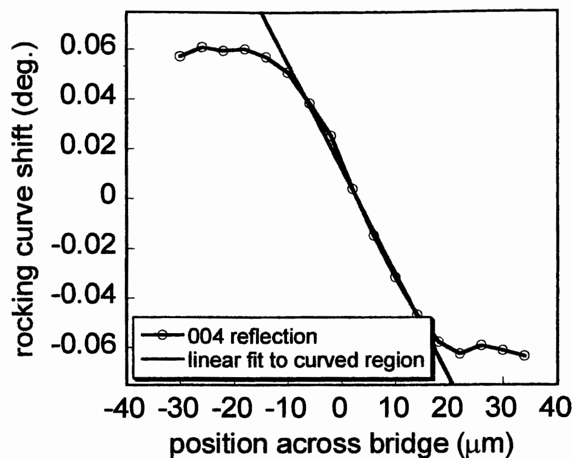

Figure 5 Distortion of microfabricated bridge beneath silicon nitride stressor layer causes a rotation of the Si atomic planes.

CONCLUSION AND OUTLOOK

The detailed control and evaluation of strain at length scales well below 1 μm that is now possible with x-ray techniques has the potential to enable a range of new devices. The manipulation of strain in micromachined silicon structures presents a new opportunity in the control of surface processes in epitaxial growth. Synchrotron x-ray microdiffraction allows simultaneous independent measurements of the strain and bending in devices fabricated for this purpose. Microdiffraction measurements show that bending is the dominant source of strain in a microfabricated Si bridge loaded at its ends by silicon nitride thin films. The total strain difference between the top and bottom of the bent bridge exceeds 0.1% in our prototype structures and can potentially be increased in optimized devices.

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