

# High resolution measurements of strain and tilt distributions in SiGe mesas using electron backscatter diffraction

Angus J. Wilkinson<sup>a)</sup>

Department of Materials, University of Oxford, Parks Road, Oxford OX1 3PH, United Kingdom

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Electron backscatter diffraction allows the elastic strain and rotation tensors to be determined at high spatial resolution and with a strain sensitivity of  $\sim 10^{-4}$ . The technique is used to investigate variations of strains and rotations near the surface of 200 nm thick epitaxial layers of  $\text{Si}_{0.85}\text{Ge}_{0.15}$  grown on a Si substrate patterned with mesa stripes. In wide mesa stripes the strain relaxation and lattice curvature are confined to the outer edges of the mesa. While in narrower mesas the relaxation extends across the entire mesa width. Measured stress levels confirm earlier predictions of the extent of relaxation with mesa width. © 2006 American Institute of Physics. [DOI: 10.1063/1.2403904]

Characterizing local strain distributions at a length scale of tens of nanometres is a considerable challenge that must be met if progress is to be made in understanding the development of stresses and strains during the production of semiconductor devices. X-ray diffraction based strain measurements cannot achieve the required spatial resolution even in third generation synchrotron facilities.<sup>1,2</sup> Micro-Raman<sup>3</sup> is also limited by spatial resolution, and the true tensor nature of the strain cannot be realized. Transmission electron microscope based techniques, such as convergent beam electron diffraction, achieve excellent spatial resolution of  $\sim 10$  nm;<sup>4</sup> however, stress relaxation within the thin foils is a major and unavoidable problem.<sup>5</sup>

Electron backscatter diffraction (EBSD) retains the high spatial resolution afforded by use of an electron probe, while avoiding the need to work with thin foils. Recent advances make it possible to map strain variations with a sensitivity of  $\sim 10^{-4}$  in a routine way.<sup>6,7</sup> Here we describe how elastic strain variations and small lattice rotations can be determined using EBSD and give some results for a  $\text{Si}_{0.85}\text{Ge}_{0.15}$  epilayer grown on a series of mesa stripes of different widths patterned on a (001) Si substrate.

A full description of the EBSD strain measurement method has been given by Wilkinson *et al.*<sup>6</sup> The EBSD system comprises a scintillator screen held in the scanning electron microscope sample chamber and viewed through a lead glass window by a  $\sim 1000 \times 1000$  pixel, peltier cooled charge coupled device camera. The screen subtends an angle of  $\sim 70^\circ$  at the sample. EBSD patterns are then recorded as the electron beam is stepped from point to point over the region of interest. Variations in the elastic strain and lattice tilts cause small shifts in the positions of zone axes and other features in the patterns. One position on the sample is selected as the reference point from which the reference pattern is obtained. Within various subregions of the pattern, small shifts of features away from their positions in the reference pattern are measured. These pattern shifts are determined using image analysis procedures detailed by Wilkinson *et al.*<sup>6</sup> To distinguish the effects of strains and tilts, it is necessary to determine the shifts in the positions at multiple subregions of the EBSD patterns. For the most general case at least four widely spaced subregions must be analyzed, but here we use

20 subregions combined with least square error methods to obtain the strain and rotation tensors that best fit the measured pattern shifts.

The sample consisted of a  $\text{Si}_{0.85}\text{Ge}_{0.15}$  epilayer grown on a (001) Si substrate patterned with shear sided raised mesas. The Si mesas were  $3 \mu\text{m}$  high with side faces oriented parallel to (110) and (1 $\bar{1}$ 0). The mesas were rectangular with length of  $400 \mu\text{m}$  along the  $[1\bar{1}0]$  direction. The widths  $w$  measured along  $[110]$  varied from  $15 \mu\text{m}$  down to  $1 \mu\text{m}$ . A 200 nm thick layer of  $\text{Si}_{0.85}\text{Ge}_{0.15}$  was then grown on top of the patterned Si. EBSD measurements were made in a JEOL JSM6500F at a beam energy of 15 keV, a beam current of  $\sim 16$  nA, and a sample tilt of  $60^\circ$ . Pattern acquisition was automated using TSL OIM DC 4 software with all patterns recorded on hard disk at full resolution for subsequent offline batchwise analysis using strain determination software

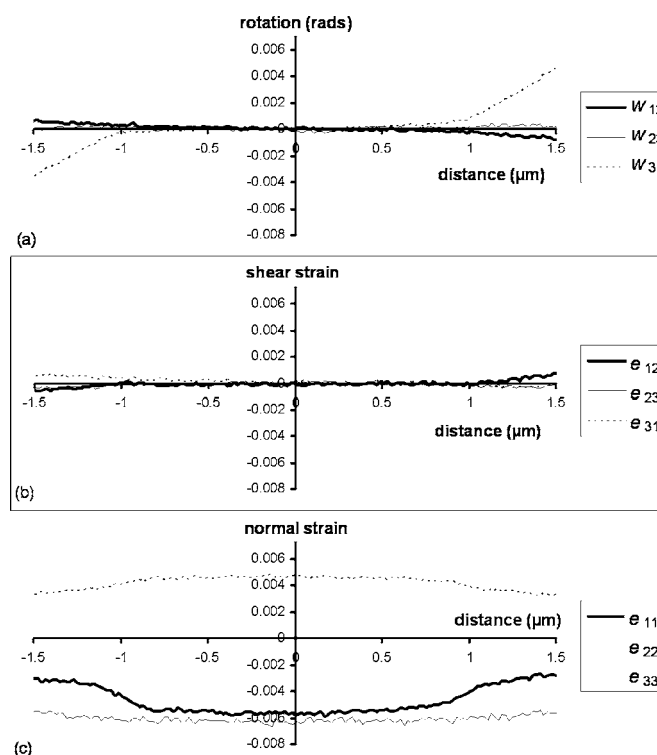


FIG. 1. Strain and rotations measured across a  $3 \mu\text{m}$  wide mesa. (a) Rotations, (b) shear strains, and (c) normal strains.

<sup>a)</sup>Electronic mail: angus.wilkinson@materials.ox.ac.uk

CROSSCOURT<sup>8</sup>. Line scans were made on plan view samples across the top surface of the mesas. The line scans had step sizes ranging from 100 nm for the widest mesa to 10 nm for the narrowest mesa. The reference pattern was obtained at the center of the largest mesa ( $w=15\text{ }\mu\text{m}$ ), where it was assumed that the strain was that of the unrelaxed epilayer (i.e.,  $e_{11}=e_{22}=-6.2\times 10^{-3}$ ,  $e_{33}=4.8\times 10^{-3}$ ). The strains will be given within the following Cartesian axes system:  $x_1$  along the width of the mesa, i.e.,  $[110]_{\text{Si}}$ ,  $x_2$  along the mesa stripe direction, i.e.,  $[\bar{1}\bar{1}0]_{\text{Si}}$ , and  $x_3$  along the wafer surface normal, i.e.,  $[001]_{\text{Si}}$ . Analysis of the EBSD patterns allows the variation in the three shear strains, and rigid body rotations about three orthogonal axes to be found directly. The analysis also gives the difference between pairs of normal strains. The three normal strains can be separated because the EBSD measurements come from very close the sample surface and should thus lead to a plane stress state. The normal stress  $\sigma_{33}$  perpendicular to the free surface must go to zero so we can write

$$\sigma_{33}=0=C_{33}e_{33}+C_{32}(e_{22}+e_{11}),$$

where  $C_{ij}$  are the elastic constants referred to the sample axes system. Specific values were those for Si:  $C_{33}=166\text{ GPa}$  and  $C_{32}=64\text{ GPa}$ .

Figure 1 shows the variations of each of the rotation and strain components across a  $3\text{ }\mu\text{m}$  wide mesa measured at 20 nm steps. There are significant rotations  $w_{13}$  about the  $x_2$  axis parallel with the long axis of the mesa [see Fig. 1(a)]. Over the outer  $\sim 0.5\text{ }\mu\text{m}$  at each mesa edge the lattice rotates as the epilayer expands outwards due to the removal of lateral constraint at the edge of the mesa. The rotations are of equal magnitude but in the opposite sense at the two mesa

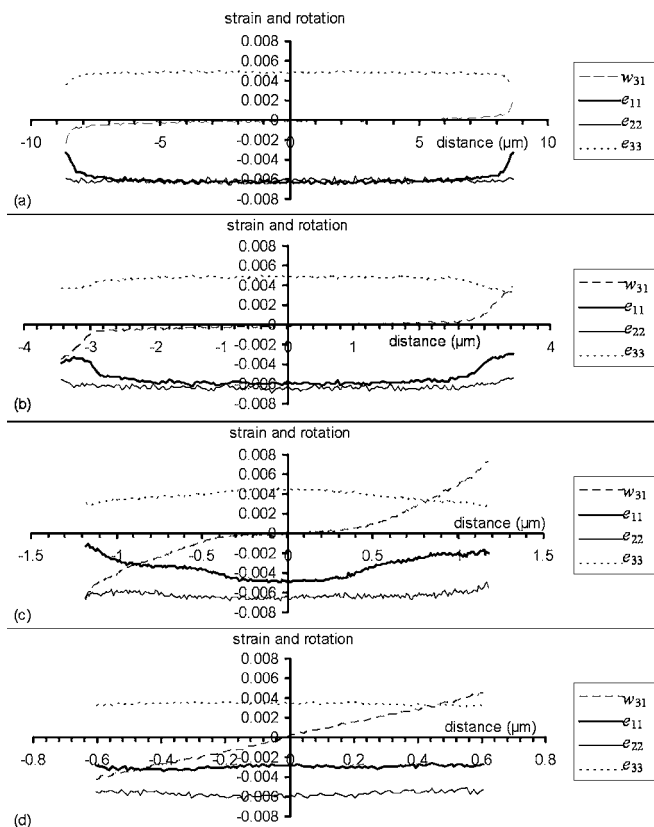


FIG. 2. Variations in most significant components of strain and rotation across mesas of different widths.

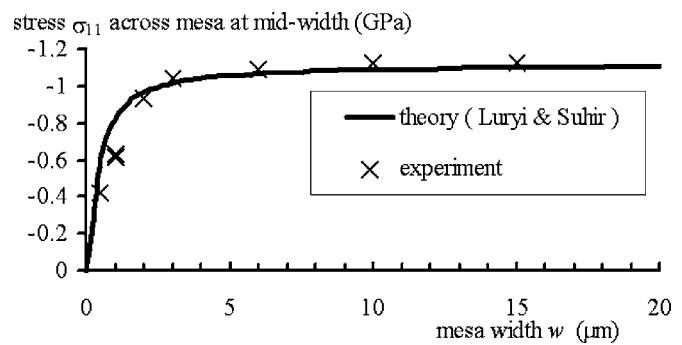


FIG. 3. Comparison between stress measured at mesa center as a function of mesa width with the theory of Luryi and Suhir (Ref. 9).

edges. There are, however, no significant rotations  $w_{23}$  and  $w_{12}$  about the  $x_1$  and  $x_3$  axes which is to be expected given the sample symmetry.

Figure 1(b) shows that there are no significant variations in any of the shear strain components, again this is as expected from equilibrium conditions and sample symmetry.

The most significant variations are in the normal strain components which are shown in Fig. 1(c). The strain  $e_{22}$  along the long axis of the mesa remains close to the unrelaxed misfit strain of  $-6.2\times 10^{-3}$  across the entire section, as expected given the long length of the mesa in that direction. The strain  $e_{11}$  acting across the width of the mesa does show significant variation. Toward the mesa center  $e_{11}$  shows slightly less compression than the  $e_{22}$  component, indicating some minor stress relaxation. However, over the outer  $\sim 0.7\text{ }\mu\text{m}$  the  $e_{11}$  strain relaxes to a significantly lower compressive strain as the epilayer expands outwards due to the removal of lateral constraint at the mesa edges. To maintain the imposed  $\sigma_{33}$  equals zero condition, the strain  $e_{33}$  takes tensile values which reduce in magnitude near the mesa edge mirroring changes in  $e_{11}$ .

The strain and rotation components of most significance are  $e_{11}$ ,  $e_{22}$ ,  $e_{33}$ , and  $w_{13}$ , and the variations of these across mesas of different widths are shown in Fig. 2. In the wider mesas studied [Figs. 2(a) and 2(b)], the normal strains were constant and unrelaxed toward the center of the mesa, while towards the edges there was a reduction in the  $e_{11}$  and  $e_{33}$  strain components. For the smaller mesa widths [Figs. 2(c) and 2(d)], the relaxation of the  $e_{11}$  strain is seen across the entire mesa width. Similarly the rotations  $w_{31}$  about the  $x_2$  axis are confined to the edges of the wider mesas, but extend across the entire width of the narrower mesas. For the narrower mesas the strain relaxation leads to much less variation in the normal strains though the lattice rotations remain.

There are several models of the stress relaxation of misfitting elastic layers grown on substrates of finite lateral width. The simple analytical model of Luryi and Suhir<sup>9</sup> is perhaps the most widely cited. Figure 3 shows the stresses  $\sigma_{11}$  at the center of the mesas as a function of the mesa width as predicted by this model. Using single crystal elastic constants the strains measured by EBSD can be converted to stress, and this is compared to the model in Fig. 3. The agreement between experiment and theory is remarkably good.

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