*Paper presented at Microsc. Semicond. Mater. Conf., Oxford, 5–8 April 1993*

# Profiling of $Ge_xSi_{1-x}$/Si strained-layer superlattices by large-angle convergent beam electron diffraction and electron holography

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**ABSTRACT:** Large-angle convergent-beam electron diffraction (LACBED) is suitable to reveal information on local strain and misfit stress relaxation of $Ge_xSi_{1-x}$/Si strained-layer superlattices (SLS) because the diffraction lines are sensitively dependent on small changes of the spacing between lattice planes. We shall demonstrate that the line shifts in a cross-sectional specimen caused by the effects of misfit strain and stress relaxation can be separated. An electron hologram taken from a $Ge_xSi_{1-x}$/Si SLS is also presented as a preliminary result.

## 1. INTRODUCTION

The band structure of $Ge_xSi_{1-x}$/Si SLSs is known to be strongly dependent on the state of strain within the epilayers. LACBED has been established as one of the most widely used techniques in materials science. The diffraction lines in the LACBED patterns can be used to investigate the strain in a lattice misfit system because their positions are sensitively dependent on a small change in lattice parameter. Since the probe is slightly defocussed, equivalent to imaging the specimen with a convergent spherical wave, a shadow image of the specimen is superimposed on a LACBED pattern in the back focal plane of the microscope. (Tanaka, Satio, Ueno and Harada, 1980, Fung, 1984, Duan, 1992a) It is important that the probe size, which defines the spatial resolution in LACBED should be as small as possible. Several workers have attempted to measure SLS misfit strains directly. In practice, the possibility of elastic relaxation of strain in the very thin samples used for cross-sectional transmission electron microscopy (XTEM) must be taken into account. In order to interpret the diffraction contrast due to the stress relaxation, some theoretical treatments of free surface stress relaxation at a misfitting interface have been carried out. (Gibson and Treacy, 1984, Perovic and Weatherly, 1991) Recently it has been shown that the relaxation in the cross-sectional specimen of $Ge_xSi_{1-x}$/Si SLS can be directly measured by the shift of the diffraction lines in LACBED. In this paper we shall demonstrate that the shifts of diffraction lines caused by strain and stress relaxation in XTEM specimen can be separated. The technique can be used to profile strain and misfit stress relaxation in the SLS. With progress in field emission gun transmission electron microscopy, there is interest in using the amplitude and phase information recorded in electron holograms. The composition and elastic strain in $Ge_xSi_{1-x}$/Si SLS can introduce phase shift which can be evaluated by electron holography. We present a preliminary electron hologram taken from $Ge_xSi_{1-x}$/Si SLS.

## 2. EXPERIMENTS

$Ge_{0.2}Si_{0.8}$(10nm)/Si(40nm) SLS was grown on (001) Si by molecular beam epitaxy (MBE). Conventional transmission electron microscopy (CTEM) observation showed that the structure was pseudomorphic with few dislocations at the superlattice-substrate interface or in the superlattice itself. The superlattice-substrate interface and the interfaces between the GeSi and Si layers appeared even and sharp. LACBED was carried out in Philips EM420 and CM12 TEMs at 100kV and a Hitachi HF-2000/FEG TEM at 200kV at room temperature. The HF-2000/FEG electron microscope can give a probe less than 1nm. The spatial resolution of the shadow image obtained is better than 2nm. The electron holography was carried out in the HF-2000/FEG TEM with a biprism between the objective lens and the intermediate lens.

## 3. RESULTS AND DISCUSSIONS

If a layer has been coherently grown on the substrate, i.e., without misfit dislocations, the strain in the growth direction can be given as $e^{\perp}=-f(1+\nu)/(1-\nu)$, where f is the misfit and ν is Poisson's ratio. For a [110] XTEM specimen, this strain will be relaxed during preparation. The relaxation along the [110] sample normal is dependent on the specimen thickness, while the strain along the $[\bar{1}10]$ interfacial direction in the plane of the sample remains unrelaxed. Thus the inclination of the lattice planes in the strained-layer to the interfacial plane will depend on both the unrelaxed strain and the relaxation σ. This makes the shift of the diffraction lines complex. If we tilt the specimen to a special zone axis, the problem may be simplified. The rotation of the reflection plane due to strain in the (001) plane and the partial relaxation along [110] was discussed by Cherns et al. (1991) and Duan (1992b). In the case of the [110] zone axis LACBED, the relaxation σ in [110] does not affect the zero order diffraction lines because σ is parallel to the reflection planes. If the sample is tilted about the [001] axis, e.g. to the [210] zone axis, the relaxation can be detected since it is no longer parallel to the reflection planes which are inclined to the interfacial plane. To investigate σ experimentally, we choose a reflection plane perpendicular to the interfacial plane, for example $(\bar{4}80)$ plane. In this case, the diffraction line $\bar{4}80$ in GeSi layer is shifted from that in the substrate. (Duan, 1992b)

Figure 1a shows a LACBED pattern of the $Ge_xSi_{1-x}$/Si SLS tilting away from the [110] zone axis a few degree about the $[\bar{1}10]$ axis. The shadow image of the SLS is clear. The diffraction lines, e.g. $\bar{2}2\bar{2}$ and $\bar{3}3\bar{3}$, near the interface and the buffer layer between the substrate and the SLS are blurred as discussed in our previous works. It is evident that the $\bar{2}2\bar{2}$ and $\bar{3}3\bar{3}$ lines are shifted in the GeSi layers from that in the Si layers by the rotation of the $(\bar{1}1\bar{1})$ reflection plane (Fig.1b). The shifts can be used to measure the unrelaxed strains in (001) plane in each GeSi layer. As expected, $\pm\bar{2}20$, $\pm\bar{4}40$ and $\pm\bar{8}80$ lines are not changed near the interface and the buffer layer between the SLS and the substrate. It is interesting that the thickness fringes of $\pm\bar{2}20$ contours are shifted in the GeSi layers. This is a composition effect since the extinction distance in GeSi layers is quite different from that in the Si layers.

Figure 2 shows the [210] LACBED patterns from a thinner area of the same specimen. The diffraction lines near the edge of the specimen are zigzagged very much. This is caused by the relaxation. As mentioned before, the diffraction lines $\pm\bar{4}80$ are very useful to measure the relaxation in [110] without the effect of the strain along [001] because the $(\bar{4}80)$ plane is parallel to [001]. Since the shift of the $\bar{4}80$ line is proportional to the relaxation σ, the curve can be used to profile the relaxation in each Si layer. The 242 lines are also very much curved. The shift of the 242 diffraction lines has two components: one corresponding to the relaxation along [110], the other to the strain along [001]. Since the latter is proportional to the tilt angle from the [210] zone axis, it is a small term near the 210 pole and then can be neglected for first approximation. Because of the presence of the thickness fringes even in very thin area, the 242 lines can give much information on the strain relaxation vs the specimen thickness.

The small probe size available on the HF-2000/FEG TEM allows us to obtain high spatial resolution in LACBED patterns since, in the limit of geometrical optics, the resolution is given by the minimum probe size. (Vincent,1989) In order to achieve high spatial resolution we need to have a small specimen defocus to increase the spatial magnification of the shadow image. This required the use of a 1μm selected area aperture specially fabricated by etching a small hole in a thin metal foil. (Vincent private communication) By minimizing the spot size to less than 1nm we can obtain results such as those illustrated in Fig.3. This shows two LACBED patterns taken near the 210 pole showing 242 and 004 contours. The spatial resolution is better than 2nm. Fig. 3a shows that the diffraction lines are evidently curved in the Si and GeSi layers with layer thickness = 40nm and 10 nm respectively. Fig. 3b shows that the presence of an interfacial dislocation splits the diffraction lines into segments in agreement with previous observations (Cherns and Preston, 1986).

Off-axis electron holography was performed in the HF-2000/FEG TEM equipped with both a cold field emission gun and a electron biprism. When a positive potential is applied to the central filament of the electron biprism located below the objective lens, the image of object and a reference beam can be made to overlap each other to form an interference pattern. Fig. 4 shows an electron hologram taken from the edge of the Si layer in the XTEM specimen of the $Ge_xSi_{1-x}/Si$ SLS. An arrow-pair indicates the edge of the specimen. In region A the hologram fringes are from free-space and in region B the lattice image is crossed by the interference fringes from which local phase and amplitude information can be extracted. (Lichte, Volkl and Scheerschmit, 1992)


## ACKNOWLEDGEMENT

X. F. Duan was supported by the exchange program between the Chinese Academy of Science and the Royal Society, U.K. and by the National Natural Science Foundation of China

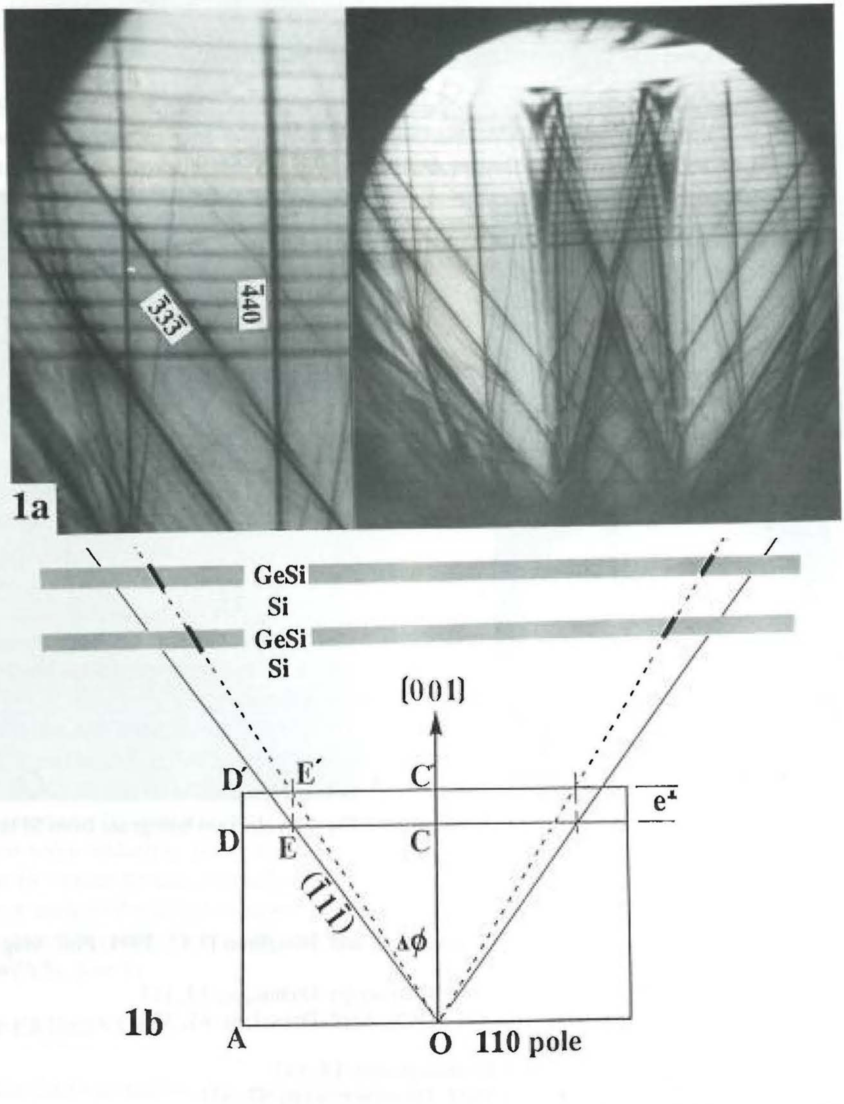




Fig.1 a [110] LACBED pattern of the $Ge_xSiB_{1-x}/Si$ SLS showing the shift of $\bar{3}3\bar{3}$ lines in GeSi layers by the strain $e^{\perp}$ along [001].

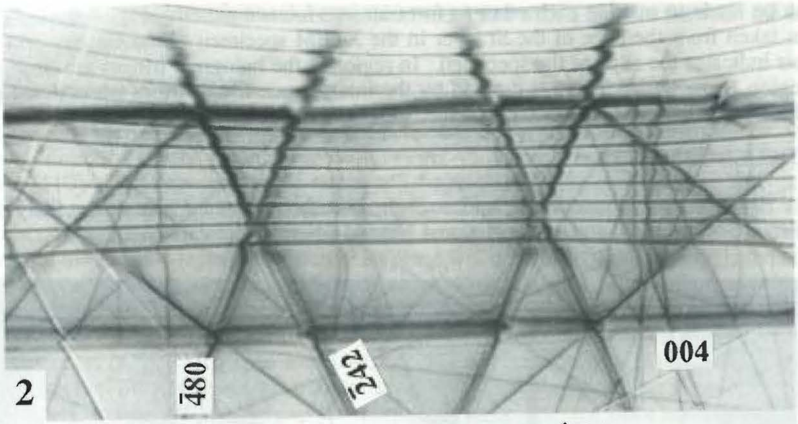
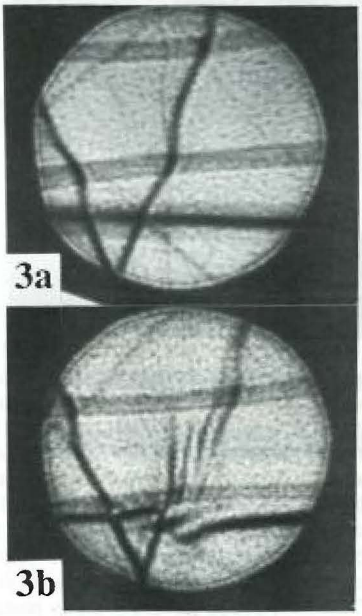



Fig.2 a [210] LACBED pattern from a thinner area of the same specimen.



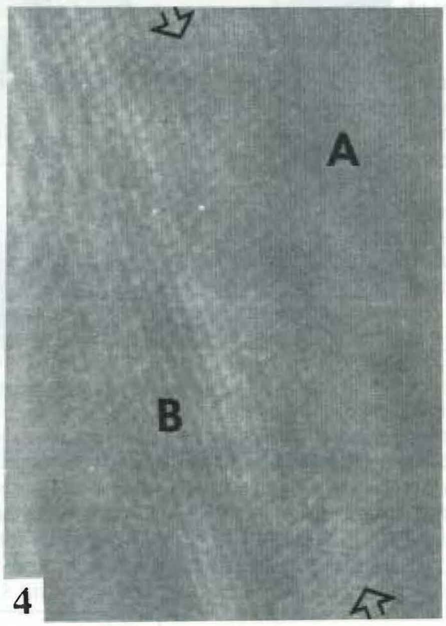




Fig.3 LACBED patterns taken using 1μm SA aperture. Fig.4 an electron hologram from Si layer.

## REFERENCES


Cherns D., Touaitia R., Preston A. R., Rossouw C. J. and Houghton D. C., 1991, Phil. Mag. **A64**, 597

Cherns D. and Preston A. R., 1989, J. Electron Microscopy Technique, **13**, 111

Duan X. F., 1992a, Ultramicroscopy **41**, 249, 1992b, Appl. Phys. Lett. **61**, 324

Fung K.K., 1984, Ultramicroscopy **12**, 243

Gibson J.M., and Treacy M.M.J., 1984, Ultramicroscopy **14**, 345

Lichte H., Volkl E. and Scheerschmit K., 1992, Ultramicroscopy **47**, 231

Perovic D. D., and Weatherly G. C., 1991, Phil. Mag. **A64**, 1

Tanaka M., Saito R., Ueno K. and Harada Y., 1980, J. Electron Microscopy **29**, 408

Vincent R., 1989, J. Electron Microscopy Technique **13**, 40